EXHIBIT
1

RESUME
ALEXANDER P. MORAVSKY

EDUCATION

Ph.D.   Chemistry, Moscow Institute for Physics and Technology (MIPT), 1975
M.Sc.   Physics of Fast Processes, MIPT, 1972
B.Sc.   Molecular and Chemical Physics, MIPT, 1970

PROFESSIONAL EXPERIENCE

1998-Present   MER Corporation, Senior Scientist, Tucson, AZ
1980-1998   Senior Research Fellow, Head of a Research Group, Institute of Problems
            of Chemical Physics, Chernogolovka, Russia
1979-1980   Post Doctorate of Prof. J.K.Stille, Colorado State Univ., Fort Collins, CO
1975-1979   Research Fellow, Institute of Chemical Physics, Chernogolovka, Russia

RECORD OF TECHNICAL ACHIEVEMENTS

- 11 Patents
- 240 papers in refereed journals
- 5 Reviews

INDUSTRIALLY IMPLEMENTED TECHNOLOGIES:

- technique for ketones and aldehydes micro-analysis
- methane absorption accumulators

AREAS OF TECHNICAL EXPERTISE

- R&D on advanced new materials, basic chemical technology, organic synthesis
- Catalysts, small molecule catalytic conversions of industrial interest
- Redox processes and fast reactions in solution and films
- Purification of substances, analytical chemistry
- Analysis of sophisticated chemical kinetic schemes
- Spectral analysis, NMR and ESR, XPS, GC, GC/MS, LC, HPLC
- Fullerene and nanocarbon materials science and technology

Dr. Moravsky A.P.
List of recent publications. 1993-2009.

1. "The Loss-Function of Solid C_{60}", Synthetic Metals, v.56, No 2-3, 2961-2966 (1993). V.I.Rubtsov, Yu.M.Shulga, A.S.Lobach, and A.P.Moravsky.

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"Photophysical and Photochemical Properties of C_{60}. Picosecond and Nanosecond Laser Photolysis Study", International Workshop on Fullerenes and Atomic Clusters", 1993, St.Peterburg, Russia, p.29. Nadtochenko V.A. et al.

"On the Reaction of Buckminsterfullerene with Tetrabuthylammonium Tetrahydroborate", Russian Chemical Bulletin, N3, 803(1993). V.D.Makhaev, Yu.M.Shulga, A.S.Lobach, V.N.Vasilets, O.S.Roshchupkina, and A.P.Moravsky.

10. "Reaction of Organic Cation Tetrahydroboronates with Fullerene C_{60}", Int. Conference "Physics and Chemistry of Fullerenes", Greece, 1993, Abstracts, p.34. V.D.Makhaev, A.S.Lobach, Yu.M Shulga. O.S.Roschupkina, and A.P.Moravsky.

"Interaction of Methane with Acetylene in the Presence of Ziegler-Natta Type Catalysts", Neftekhimiya, v.32, N4, 324-330 (1992). E.M.Efimova, A.P.Moravskii, and N.F.Noskova.

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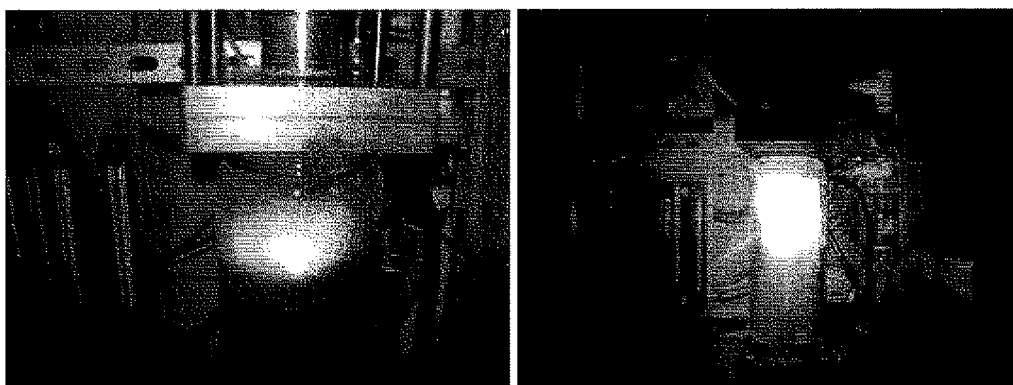
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EXHIBIT
2
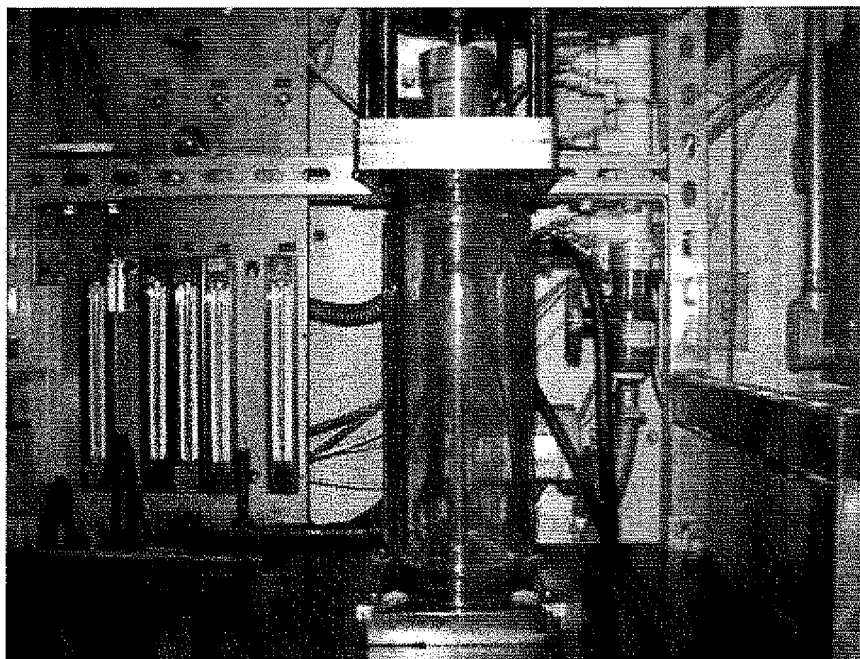
Quartz chamber with carbon-rod electrodes 1 cm in diameter, before the arc operation

Quartz chamber with 1 cm diameter carbon-rod electrodes, during the direct current arc operation at 200 A in 100 Torr Argon gas atmosphere (left), and in 10 Torr Argon gas atmosphere (right).

EXHIBIT
3
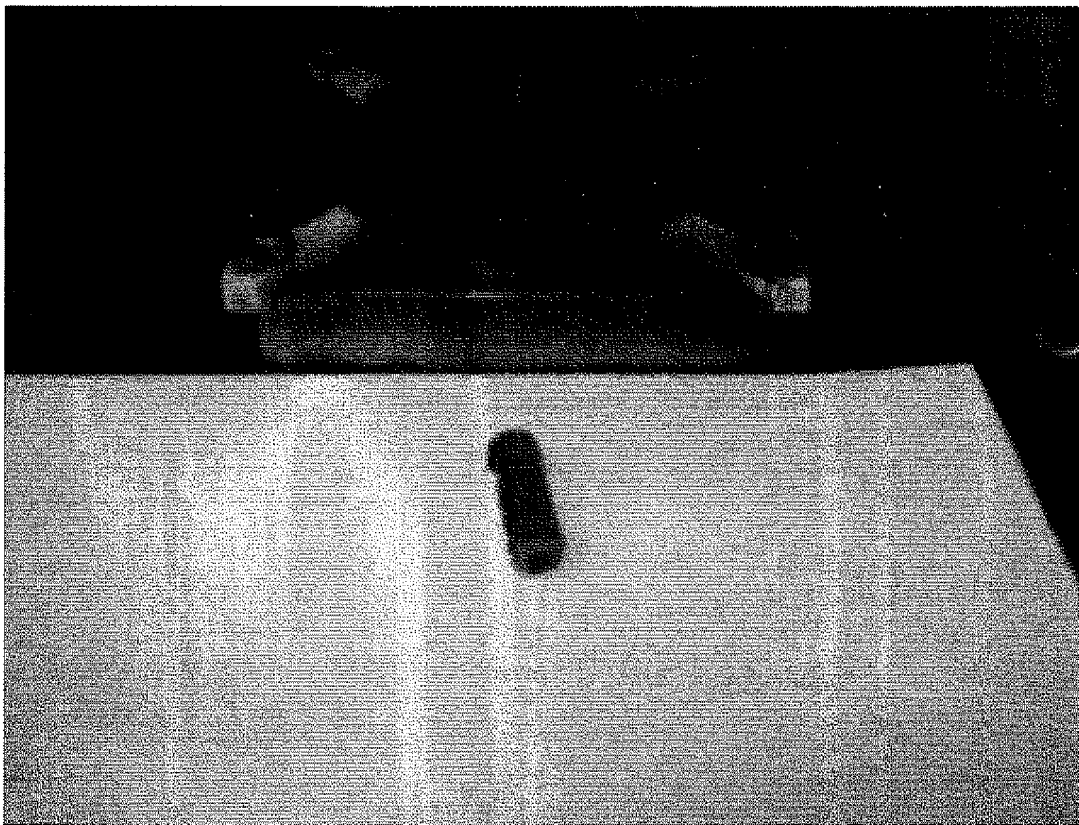
Cathode deposit formed in the arc process conducted at 200 A direct current and 100 Torr argon gas pressure

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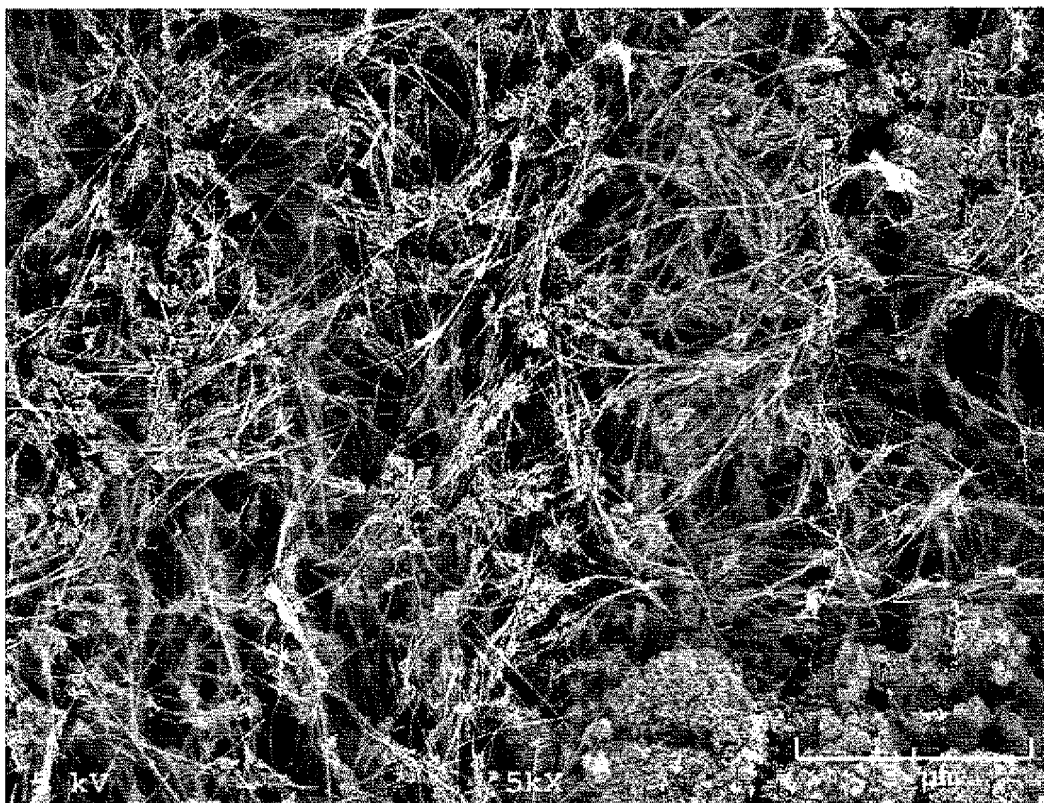
Low magnification SEM image of the carbon nanotube containing product from the arc synthesis performed at 100 Torr argon gas pressure (Run "Arc #1")

EXHIBIT
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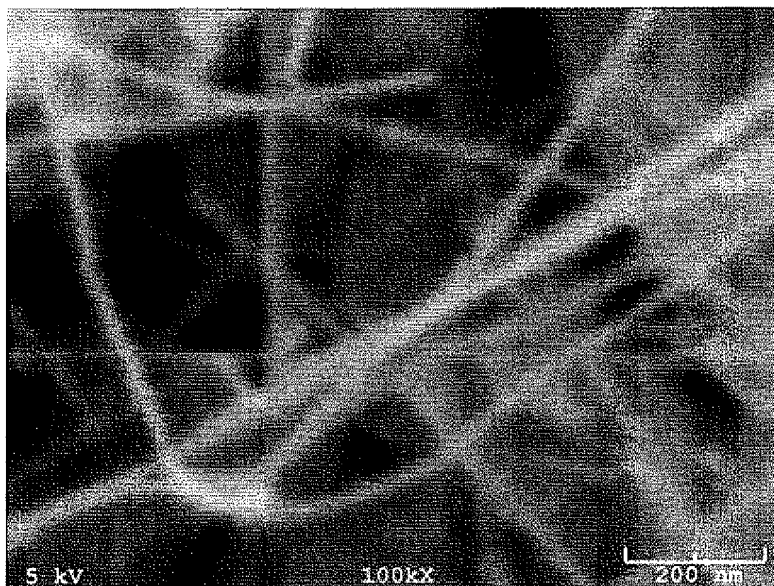
High magnification SEM image of carbon nanotubes produced in the run "Arc #1" in 100 Torr argon gas atmosphere.
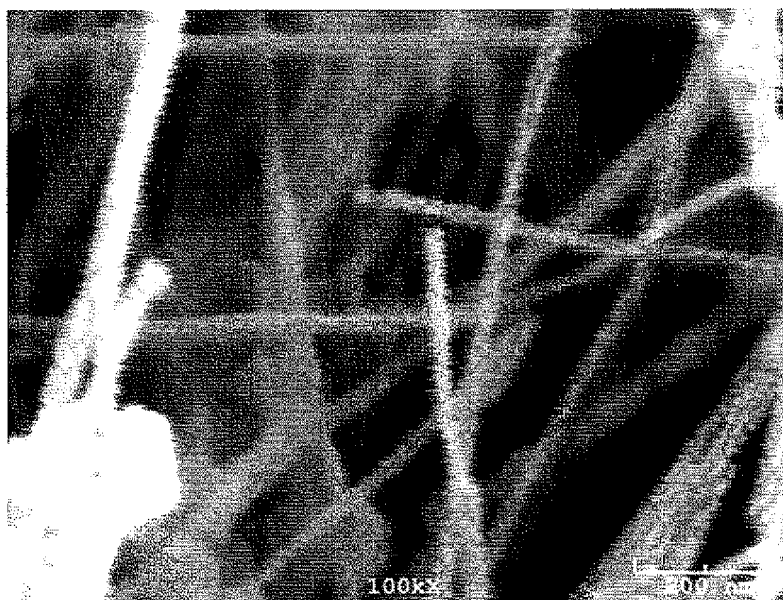
High magnification SEM image of carbon nanotubes produced in the run "Arc #1" in 100 Torr argon gas atmosphere. Gold-coated sample.

EXHIBIT
6
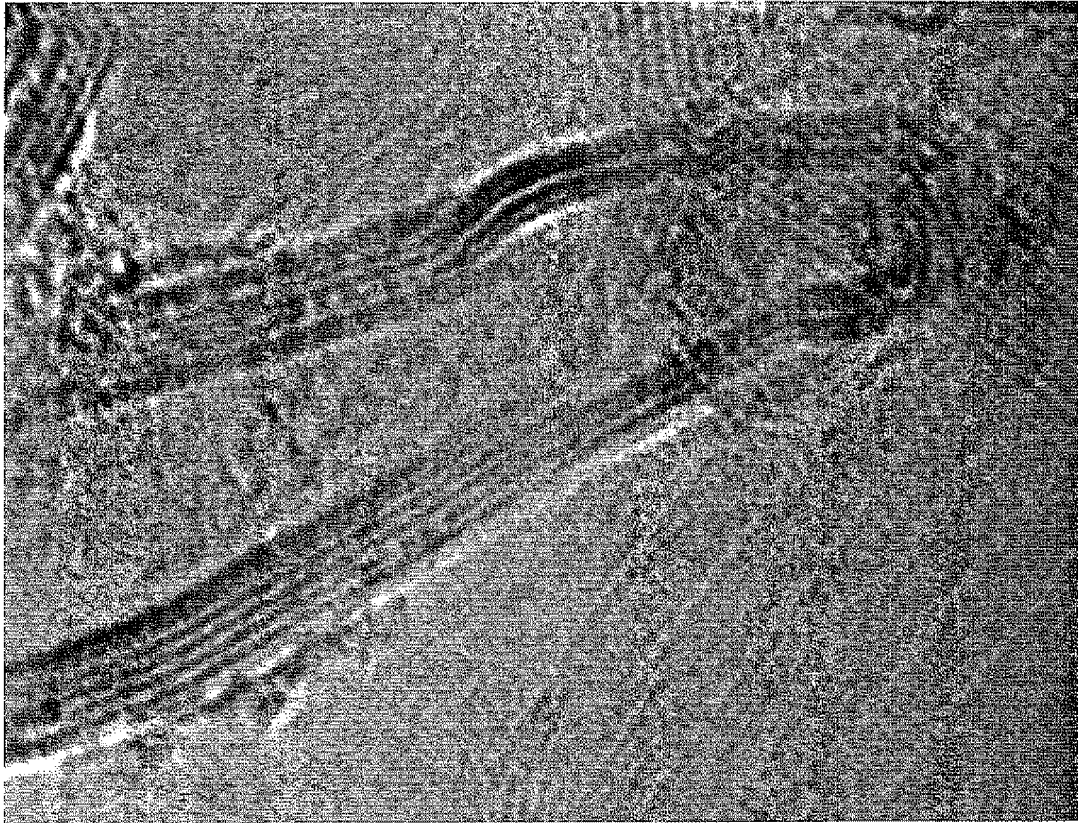
HRTEM image of a 7-wall MWNT that transforms in a 4-wall MWNT near the tube end. Outer Diameter varies from ~ 5 to ~8 nm. Arc #1. Image size 26.7nm x19.9nm.

HRTEM image of a 3-wall end fragment of a MWNT. Outer Diameter at the end ~ 5 nm. Run Arc#1. Image size 62.0x27.3nm.

EXHIBIT
7
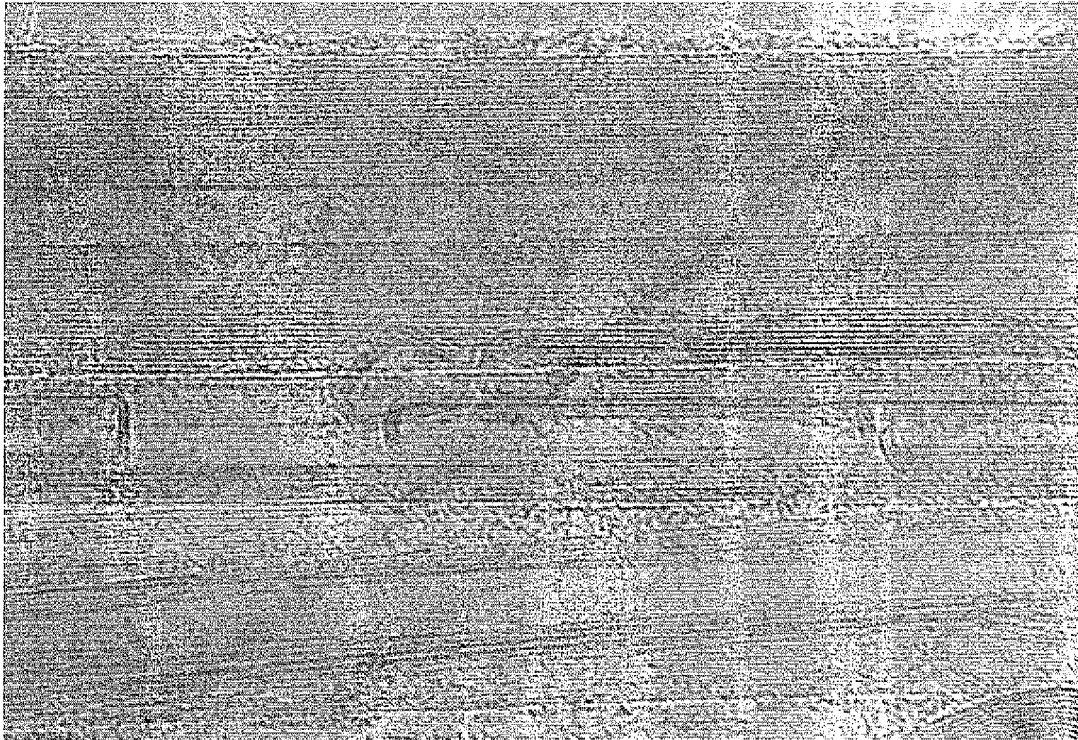
HRTEM image of a 4-wall MWNT with o.d. ~ 6 nm and 5-wall MWNT with o.d. ~ 8 nm superimposed nearly parallel with thick MWNTs: 23-wall with 22 nm o.d., 15-wall with 16 nm o.d., 7-wall with 8.2 nm o.d. Arc#1. Image size 74x50 nm.

HRTEM image of a 4-wall MWNT with o.d. ~ 3.5. Arc #1. Image size 84x13 nm.

HRTEM image of a 5-wall MWNT with o.d. ~ 6.8 nm. Arc #1. Image size 30x8.8 nm.

EXHBIT
8
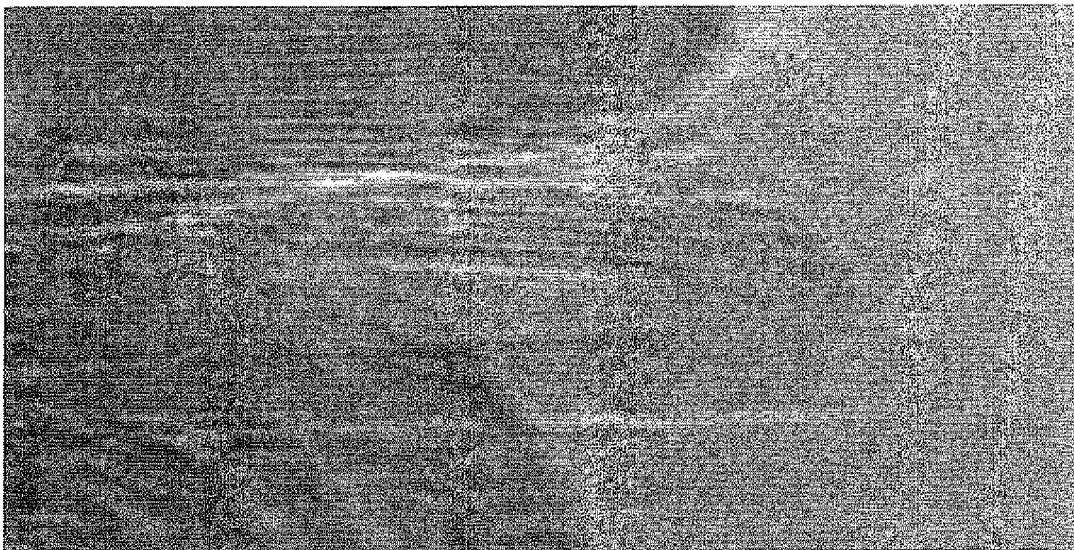
HRTEM image of the end of a 7-wall MWNT with o.d. ~ 7 nm. Image size 32x16nm.
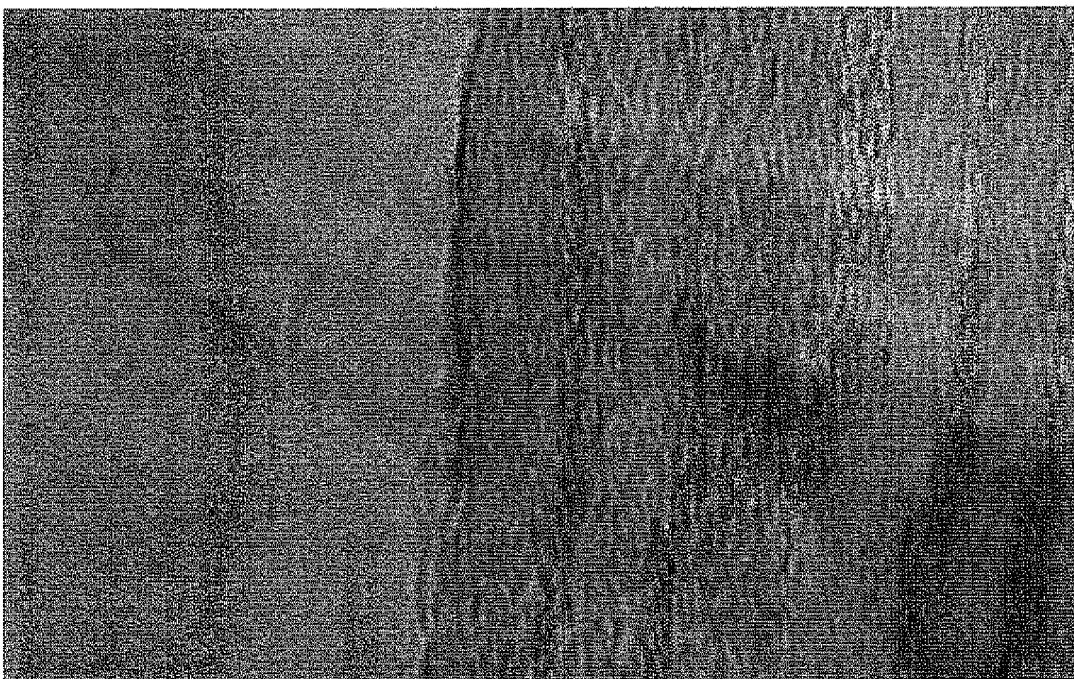
HRTEM image of conical ends of MWNT s with o.d. of caps ~ 6 nm. Arc#1. 58x35nm.

HRTEM image of a 6-wall MWNT with o.d. ~ 10.4nm and 10-wall MWNT with o.d. ~ 11.7 nm. Arc#1. Image size 205x34nm.

EXHIBIT
9

HRTEM image of 8-wall MWNT with o.d. ~ 11nm. Arc#1. Image size 91x24nm.

HRTEM of a 8-wall MWNT of 7.8 nm outer diameter, 9-wall MWNT (o.d. 8.5 nm), and 16 wall MWNT of 15.3 nm outer diameter. Image size 89.3x42.8 nm.
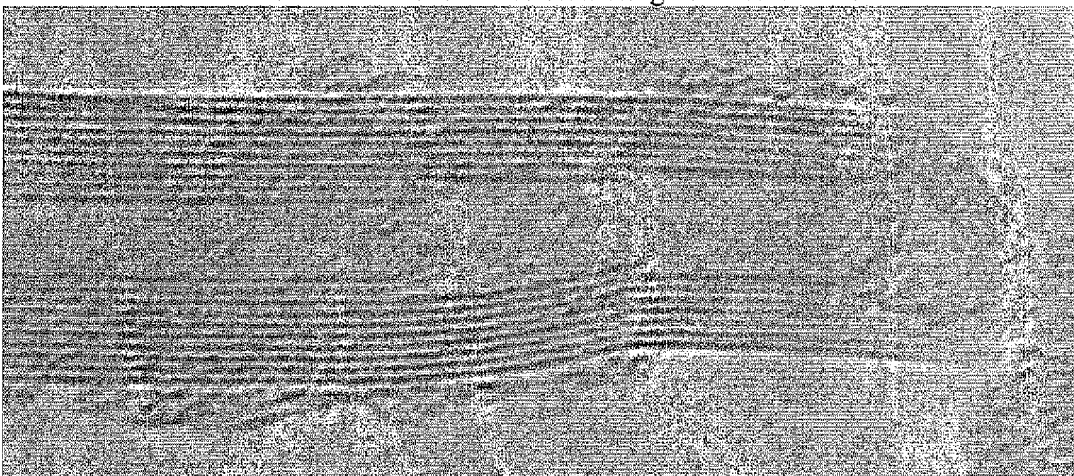
HRTEM image of a 10-wall MWNT with o.d. ~ 10nm that transforms in 7-wall MWNT with o.d. ~8.4 nm, and then in 5-wall cap. Arc#1. Image size 38x16.6nm.

EXHIBIT
10

HRTEM image of 11-wall MWNT with o.d. ~ 14nm that transforms in 5-wall MWNT with o.d. ~8.2 nm, and then in 3-wall cap with o.d.~ 6 nm. Arc#1. Image size 113x27nm.
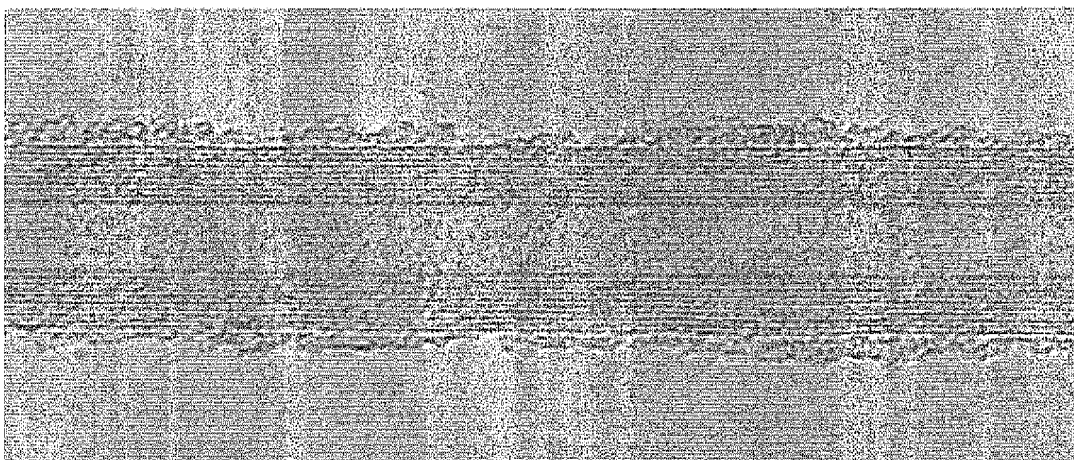
HRTEM image of a 10-wall MWNT with o.d. ~ 11.5 nm. Arc#1. Image size 70x28.6nm.
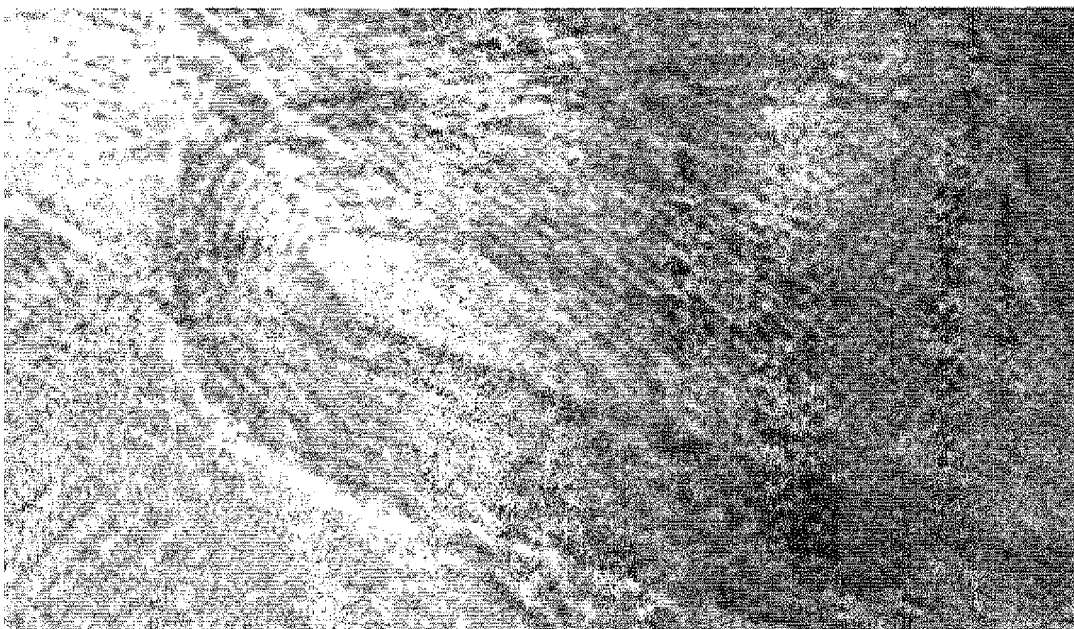
HRTEM image of a10-wall MWNT with closed tube end. O.D. ~ 10 nm. Arc#1. Image size 26.7nm x15.5nm.

EXHIBIT
11
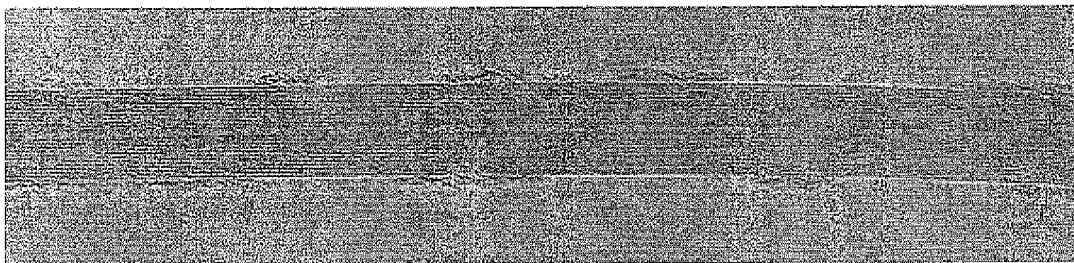
HRTEM of a 12-wall MWNT with o.d. 9.9 nm. Image size 120x28.2 nm.

HRTEM of a 14-wall MWNT with o.d. 12 nm. Image size 90.8x20 nm.

HRTEM of a 14-WNT, o.d. 15 nm; and 26-WNT, o.d. 22.5 nm. Image size 83.3x41.4 nm.

HRTEM of a 15-wall MWNT with o.d. 16nm, and a SWNT formed by cross-section partitions inside the MWNT channel with inner diameter of 4.8 nm. Image108x18 nm.
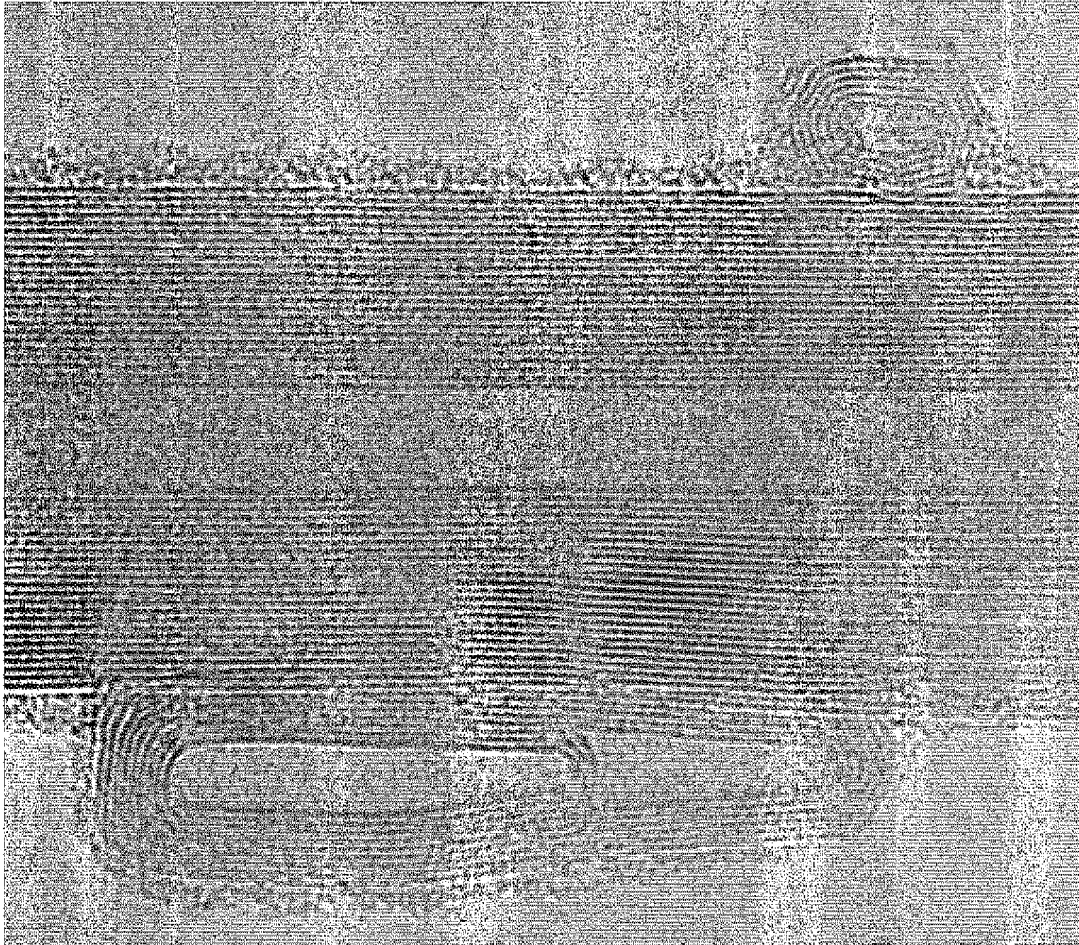
EXHIBIT 12
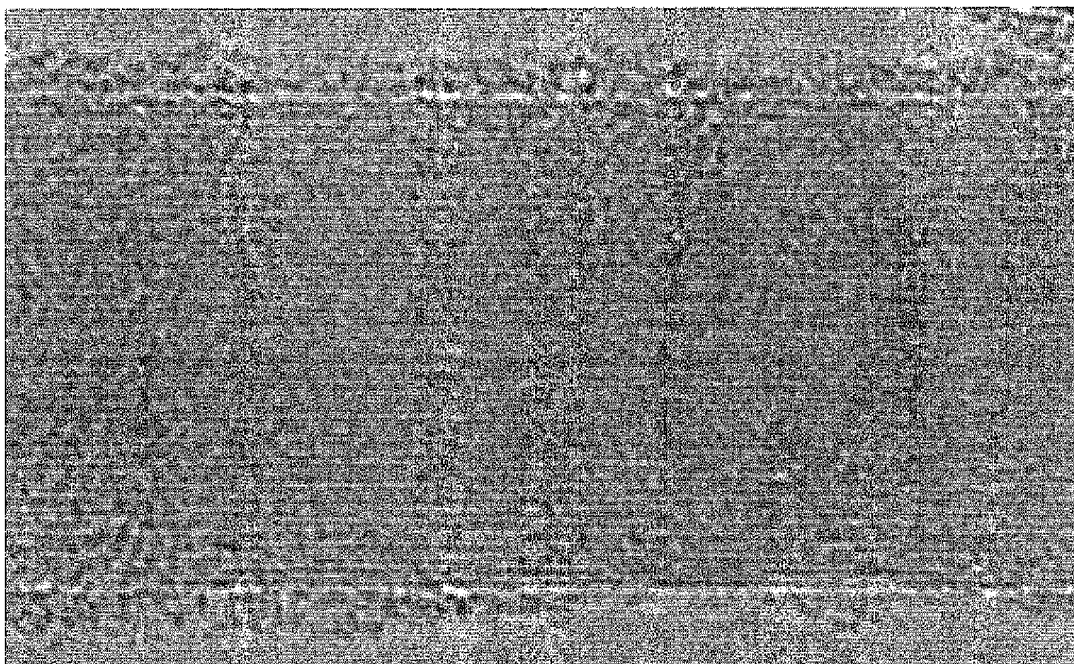
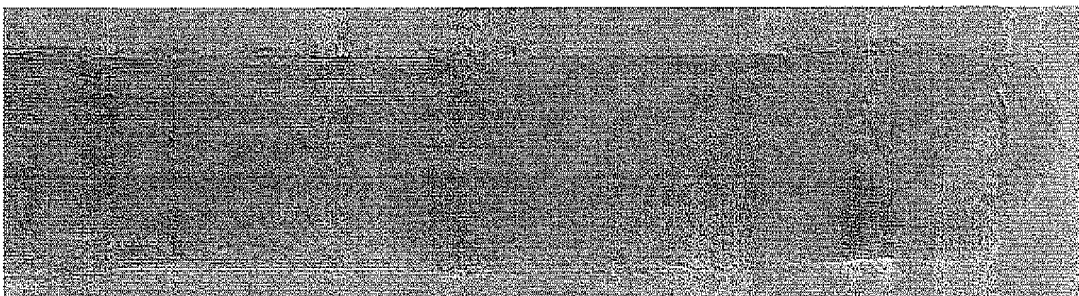HRTEM of a 20-wall MWNT with o.d. 16.4 nm. Image size 37.1x22.23 nm.
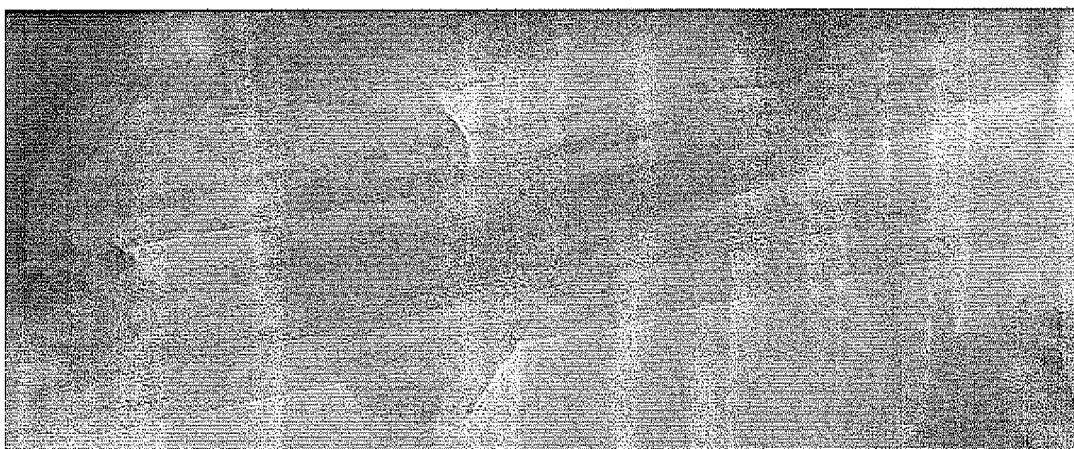
HRTEM image of MWNTs with o.d. ~ 23; 25; 30, 44; 47.7 nm. Arc#1. Image size 200x81nm.
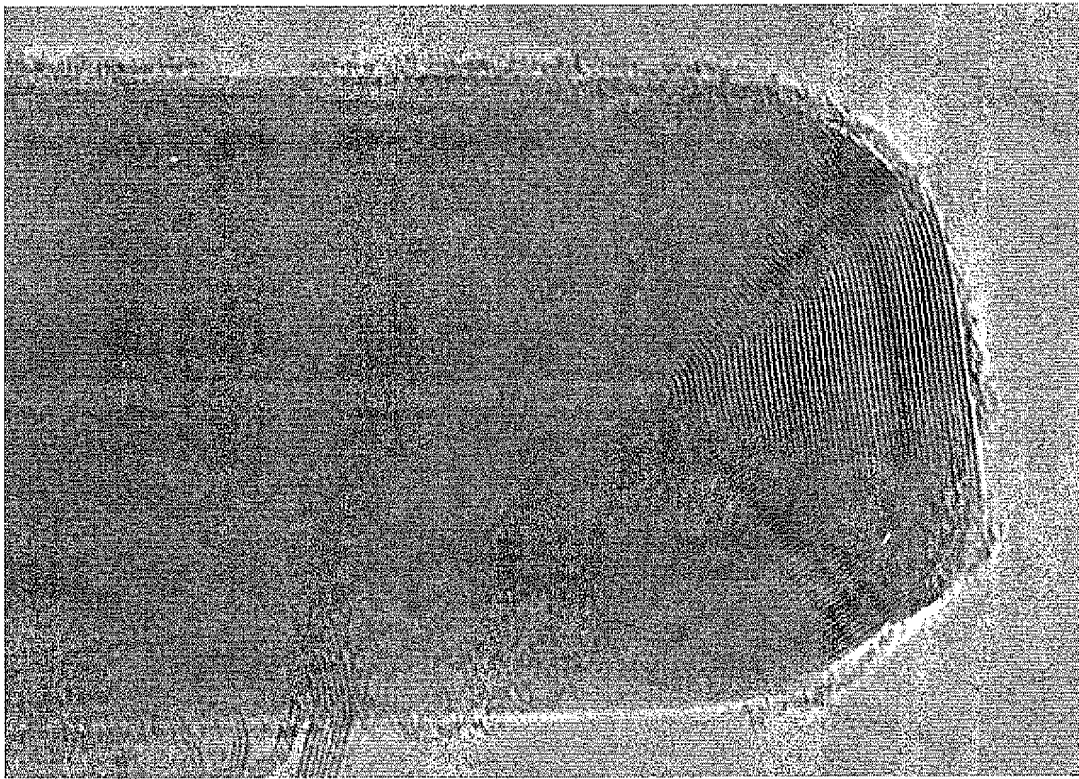
EXHIBIT
13

HRTEM of a 24-wall MWNT with o.d. ~ 22.7 nm with Multiwall Polyhedral Particles (MPPs)
attached to the wall. Arc#1. Image size 51x43 nm.

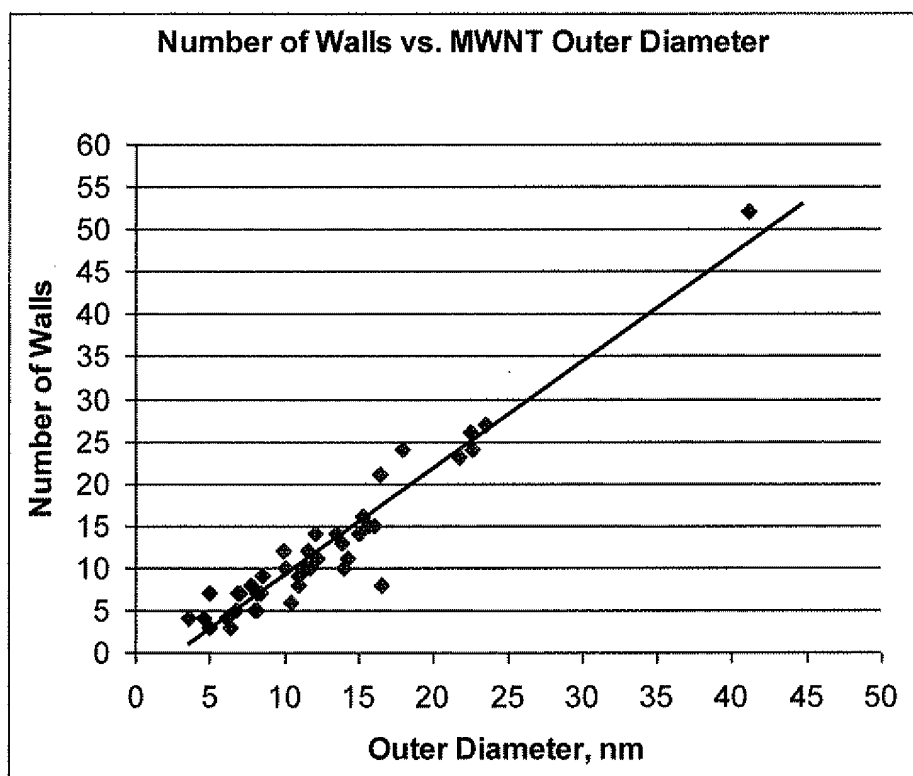HRTEM image of 27-wall MWNT with o.d. ~ 24nm. Arc#1. Image size119x32nm.

EXHIBIT
14

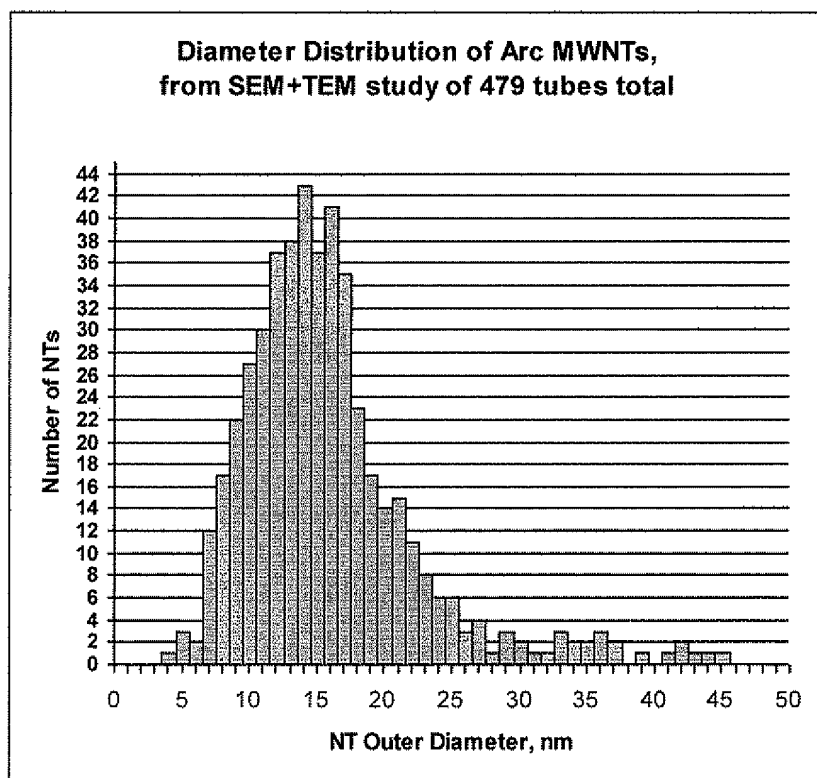HRTEM of a 52-wall MWNT with o.d. 41 nm. Image size 70x49.5 nm.

EXHIBIT
15

Number of Walls vs. MWNT Outer Diameter

Number of walls in MWNTs grows approximately linearly with the outer diameter of MWNTs, as determined from HRTEM measurements.

EXHIBIT
16

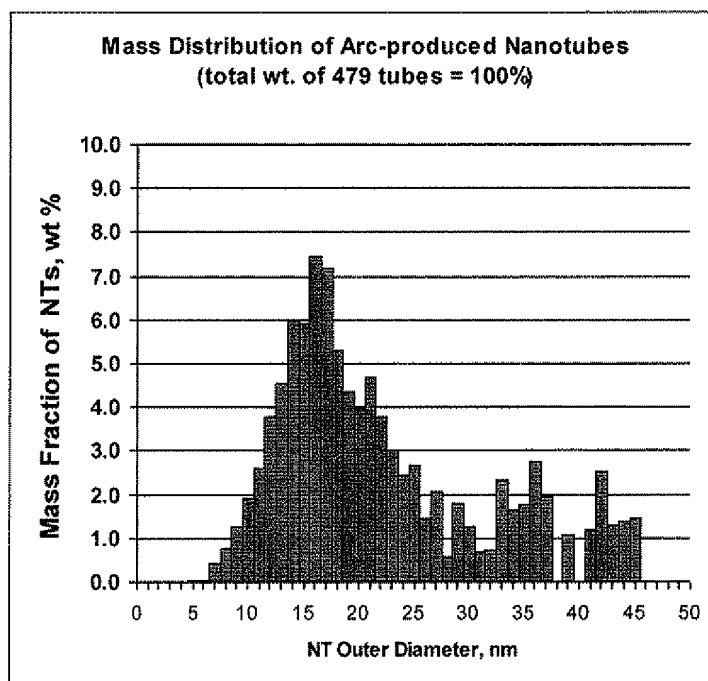**Diameter Distribution of Arc MWNTs, from SEM+TEM study of 479 tubes total**
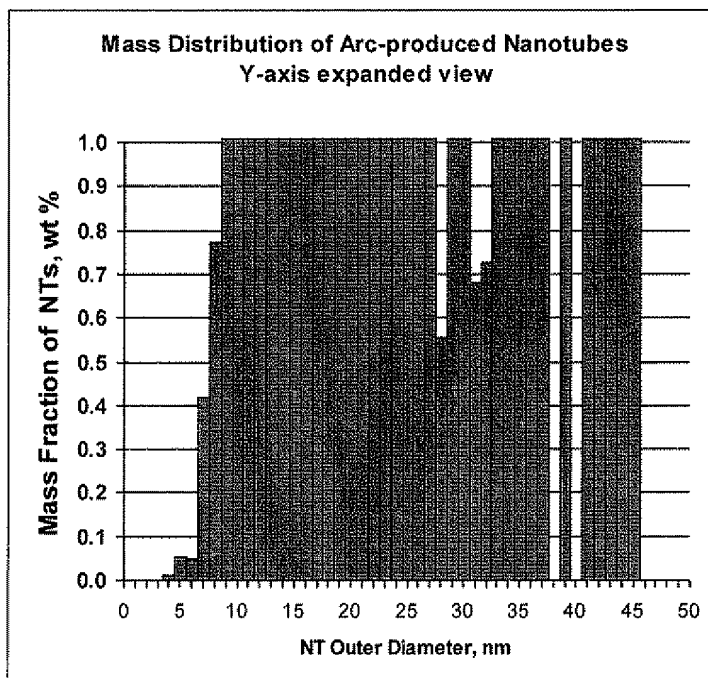
Outside diameter distribution of carbon nanotubes produced in the arc synthesis conducted under conditions of "326"

EXHIBIT 17

Mass density distribution over the outside diameter of arc-produced nanotubes

Expanded view of mass density distribution

EXHIBIT
18

SEM and HREM study of the internal structure of nanotube rich carbon arc cathodic deposits

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Abstract

The structural components and the internal organization of carbon cathodic deposits fabricated using an arc with the conditions adjusted for the effective production of nanotubes have been characterized by transmission electron microscopy (TEM). high resolution electron microscopy (HREM) and scanning electron microscopy (SEM). Typically. such deposits are columnar structures oriented along the growth direction. Three main components were observed: multiwalled nanotubes. multilayer polyhedral particles and curved graphitic formations. The measured distributions and relative quantities of the components depended on the deposition regimes. Column interiors were composed of a mixture of all these components. while the outer covering of the columns was formed predominantly of nanotubes. Multiwalled nanotubes formed a mesh-like arrangement around these columns and their growth surfaces, with smaller amounts of nanotubes present inside the columns. Nanotubes in various fragments of the deposits were mainly oriented at high angles to the deposit axes. In the column coverings. groups of tubes oriented at angles $>45°$ to the axes were present with no sets of nanotubes or bundles aligned along the deposit axes seen. Finally. a mechanism of deposit formation is proposed in connection with the recorded data. © 1999 Published by Elsevier Science Ltd. All rights reserved.

1. Introduction

Arc-discharge cathodic deposits can be regarded as a source of multiwalled carbon nanotubes (MWNs) — a novel material of solid state science that has been identified in a number of applications. for example as efficient emitters of electrons [1,2].

Though information on the internal structure of cathodic deposits that comprise of substantial amounts of MWNs can be found in various publications [3–13]. still the data from direct observations are not complete enough to understand and to solve many technological problems. These include the synthesis of uniform MWNs of a particular type. their extraction in large quantities from the deposit material. purification of the MWNs from accom-

panying components (graphitic particles and multi-layer polyhedral particles; MPPs). and a definitive quantitative analysis of the deposit contents.

Besides this. the internal structure of the deposit is the most descriptive source of information about the processes that take place in the arc and at the growth surface. and thus it forms the basis for theories on the mechanisms of formation of MWNs and MPPs. An improved understanding of the mechanism of nanotube growth can be attained from the data on the internal structure of the deposits versus arc characteristics.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are very informative techniques for the direct investigation of the internal structure and composition of a cathodic deposit. With the help of these methods. it has been established that under appropriate arc conditions for nanotube synthesis, the core of a deposit has the highly ordered structure of long (up to several mm) parallel columns of about 50 μm in diameter.

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The tops of these columns, that are the growth surfaces, play an important role in the electric arc process, in particular by being (according to [6]) the principal area of electron emission into the arc plasma. It becomes clear that such details of the deposit structure as the direction of nanotube orientation should be of prime importance for understanding the nature of some macrocharacteristics of the deposit, e.g. its magnetic properties [5,13]. As the structural details are most reliably found by direct observation, SEM investigations of columns and their tops have received most of our attention in this work. The principal aim of the SEM investigations was to understand the localization and orientation of nanotubes and nanotube bundles in the deposits. Particular attention was focused on the search for sets of nanotubes that were oriented in the same direction and especially aligned along the axis of the deposit growth. In our work we used methods of sample preparation for the SEM investigations that minimized the damage to the original structure.

Nanotube aggregates and their size distributions have been explored by conventional transmission electron microscopy (TEM). The internal structures of nanotubes and MPPs have been studied in greater detail by high resolution electron microscopy (HREM).

It has been found in the present work that structures of columns and their tops are different from other reports (for example in [6]) based on either indirect data, or on SEM investigations of samples prepared by methods that can alter the original structure.

2. Experimental

2.1. Preparation of cathodic deposits

Deposits have been fabricated by the DC arc method adjusted with the help of electron microscopy to give a high yield of nanotubes. The lower vertical graphite anode was in most experiments 6 mm in diameter and 200 mm long. Thicker graphite anodes (~10 mm) were used as well. The upper stepping-motor driven graphite cathode of 15 mm in diameter was the site of a downward growing cylindrical deposit. Each run was conducted under three fixed parameters: (1) the rate of feed of the graphite rod into the arcing zone, (2) the helium pressure and (3) the arc current. Fluctuations of the latter were monitored with an oscilloscope so as to adjust the stable regime of the discharge, which has been recommended for obtaining high quality tubes [3–6]. The reaction chamber was water cooled.

These three externally controlled parameters define the values of two observed derivative parameters of the arc process, namely the gap width and the voltage drop across the gap. These parameters are interdependent (as discussed later), so that any variation of the controlled parameters changes the derivative parameters. In the present search for better technology of nanotube production it is reasonable

to relate the obtained results, including the above mentioned derivative parameters, to a given set of the named three externally controlled parameters, ensuring the possibility of reproducing the attained experimental results.

Under conditions used in this work the deposits obtained had the form of cylinders 50 to 100 mm long and 5–12 mm in diameter. As it could be seen by the naked eye or in the optical microscope at low magnification, the hard outer grey shell of a cylinder enveloped a soft black fibrous core containing (according to electron microscopy studies described later) nanotubes of various forms, their aggregates, nanoparticles and various forms of graphite. The concentration of nanotubes in the core of investigated deposits depended on the deposition conditions and varied over a wide range from almost entirely absent (several tubes or their bundles in the SEM field of view ~25 μm^2 in size) up to the level where some regions of deposits consisted predominantly of tubes (~100–200 tubes in the same field of view).

Both untreated cathodic deposits (in preference) and deposits subjected to oxidation in air [6] and/or acid solutions of potassium permanganate [7] were investigated. The oxidation processing was aimed at increasing the nanotube concentration in the deposits through the removal of other components, which can otherwise hide the tubes from view.

2.2. Electron microscopy measurements

Specimens for TEM and HREM were prepared by dispersing the core parts of deposits in acetone by using an ultrasonic bath. A drop of the suspension was put on a lacy carbon film supported by a copper grid. Specimens were investigated in a Philips EM-430ST electron microscope operated at 200 or 250 keV, and in a Jeol JEM 4000EX microscope operated at 400 keV. Several tens of low magnification micrographs of each specimen were made for semiquantitative characterization of a deposit. The most typical structural components were investigated further by HREM in phase contrast mode at a point-to-point resolution ~0.16 nm.

For SEM investigations, deposit growth surfaces, cross-sections, and randomly split-apart fragments, both as received and after oxidation treatment, were considered. They were studied at various angles by using a low-voltage Jeol JSM-840 microscope. Deposit rods were split by manually holding the sample away from the area to be investigated. Care was taken not to disturb these freshly exposed areas. Usually the appearance of cross-sections was step-like with transverse and longitudinal (with respect to the deposit axis) regions, with columnar structures clearly visible in the core of a deposit. No special attempts were made to get longitudinally split columns, as the high mechanical stresses that would be used in these cases could alter their internal structure. Samples were fixed to SEM holders with silver dotite paint applied to the samples

No sample coating was used.

The JSM-840 microscope was also operated in high resolution mode with the accelerating voltage at 12 keV and the electron beam current ~10 pA. Secondary electron images were recorded at working distances of about 15 mm. The low values of the beam current provided the necessary spatial resolution and eliminated the possibility of electron-beam modification of the samples during observation.

3. Results

3.1. HREM and TEM of deposit components

Three distinct structural components have been observed in all the investigated specimens: MWNs of the type first observed by Iijima [8], MPPs, and various kinds of graphitic particles. A HREM image of such MWNs and MPPs is shown in Fig. 1. Along with approximately isometric particles, formations intermediate of tubes and MPPs have been observed in small quantities, for example elongated particles constructed of two to four sets of conical layers. Some varieties of MPPs and MWNs seem

elongated MPPs that terminate as cylindrical nanotubes. Graphitic components have been mainly found as coiled graphite ribbons (Fig. 2). The quantities of these components in the deposits as well as the tube diameters and sizes of the MPPs depend very much on the arc parameters.

The majority of investigated specimens could be divided into three groups according to the controlled parameters: (1) fabricated at different He pressures and arc currents, while keeping the linear feed velocity constant: this group is exemplified by the first three entries in Table 1 (specimens 1–3); (2) different only in feed velocity (specimens 4 and 5); (3) fabricated at high He pressures (specimens 6 and 7). Several thicker deposits ~11 mm in diameter produced with thicker graphite anodes were also prepared for structural investigations (specimen 8).

Distributions of nanotube diameters and linear sizes of isometric multilayer polyhedral particles have been found by TEM observations. Corresponding histograms are drawn in Fig. 3 and brief descriptions of sample compositions derived from TEM investigations are given in Table 1. Specimens produced at low He pressure contained small amounts of tubes. Thus the main component of specimen 1 was curved graphite (Fig. 2). The tendencies for the tube quantity to increase and for the graphitic components to decrease with the increase in He pressure and the decrease in arc current are clearly revealed by the first three examples in Table 1. This statement is confirmed by investigations of deposits obtained at higher pressures. Thus in specimen 6, the main components were tubes and
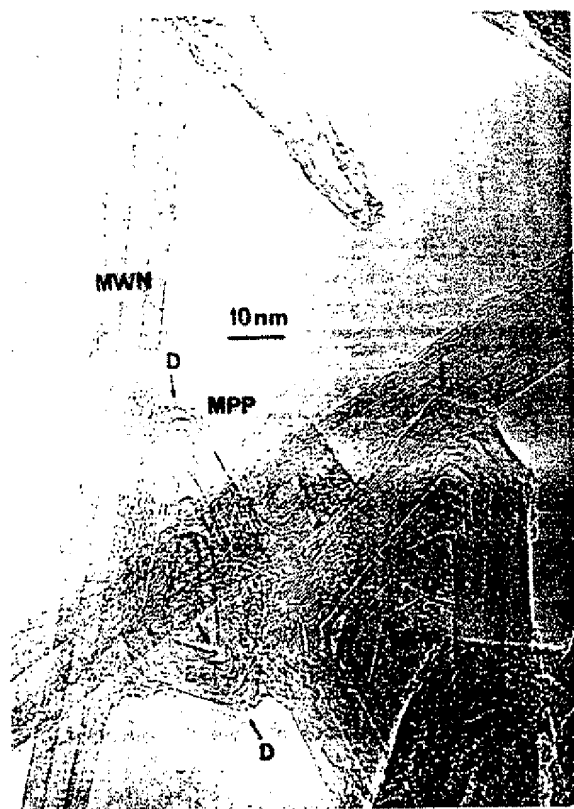

Fig. 1. HREM image (400 keV) of multilayer polyhedral particles (MPPs) and multiwalled nanotubes (MWNs). Inner cavities and defects in vertices (D) are visible. Specimen 3 (for fabrication parameters see Table 1).

Fig. 2. HREM image (400 keV) of specimen 1 that comprised of small amounts of nanotubes (MWN) and onionites (O). Curved graphite (G) was the main component of the specimen.

Table 1
Conditions and parameters of the arc process during the production of the studied deposits

Sample number	Graphite rod diameter (mm)	Deposit diameter (mm)	Helium pressure(torr)[a]	Arc current(A)	Anode feed velocity(mm/min)	Gap width(mm)	Voltage drop across the gap (V)	Deposit growth rate (mg/min)	Brief description of the deposit composition derived out of the electron microscopy observations
1	6	7.8	100	80	4	3.9	18.0	115	Low tube concentration; main component — curved graphite (Fig. 2); MPPs had rounded vertices and resembled onion-like particles
2	6	6.5	200	65	4	2.2	19.0	170	More tubes and MPPs than in sample 1; tube diameters 8–22 nm, the most probable diameter — 15 nm (Fig. 3a); the size of MPPs — 20–85 nm (Fig. 3c)
3	6	7.4	700	55	4	0.5	20.8	260	The lowest amount of graphite in the group of samples 1–3; tube diameters — 8–23 nm, the most probable diameter — 12–14 nm (Fig. 3a)
4	6	8.0	500	65	8	0.4	21.3	350	Tubes 5–29 nm in diameter (Fig. 3b); MPPs 19–93 nm in size (Fig. 3d); many graphitic particles
5	6	7.6	500	65	2.5	3.5	22.3	90	Tubes 5–46 nm in diameter; the most probable diameter 14–16 nm (Fig. 3b); MPPs 9–93 nm in size, the most probable size — 27–49 nm (Fig. 3d); very low amount of graphite
6	6	5.6	1500	65	1.4	4.2	31.1	20	The main components — tubes and MPPs (Fig. 4); tube diameters — 4–60 nm, (Fig. 3a); the sizes of MPPs — 8–120 nm (Fig. 3c)
7	6	5.5	2000	65	1.4	3.3	32.1	22	Composition is close to that of sample 6. High tube concentration, MPPs and graphitic particles
8	10.6	11.3	500	160	0.65	0.8	22.4	160	Composition is close to that of sample 6. High tube concentration, MPPs and graphitic particles

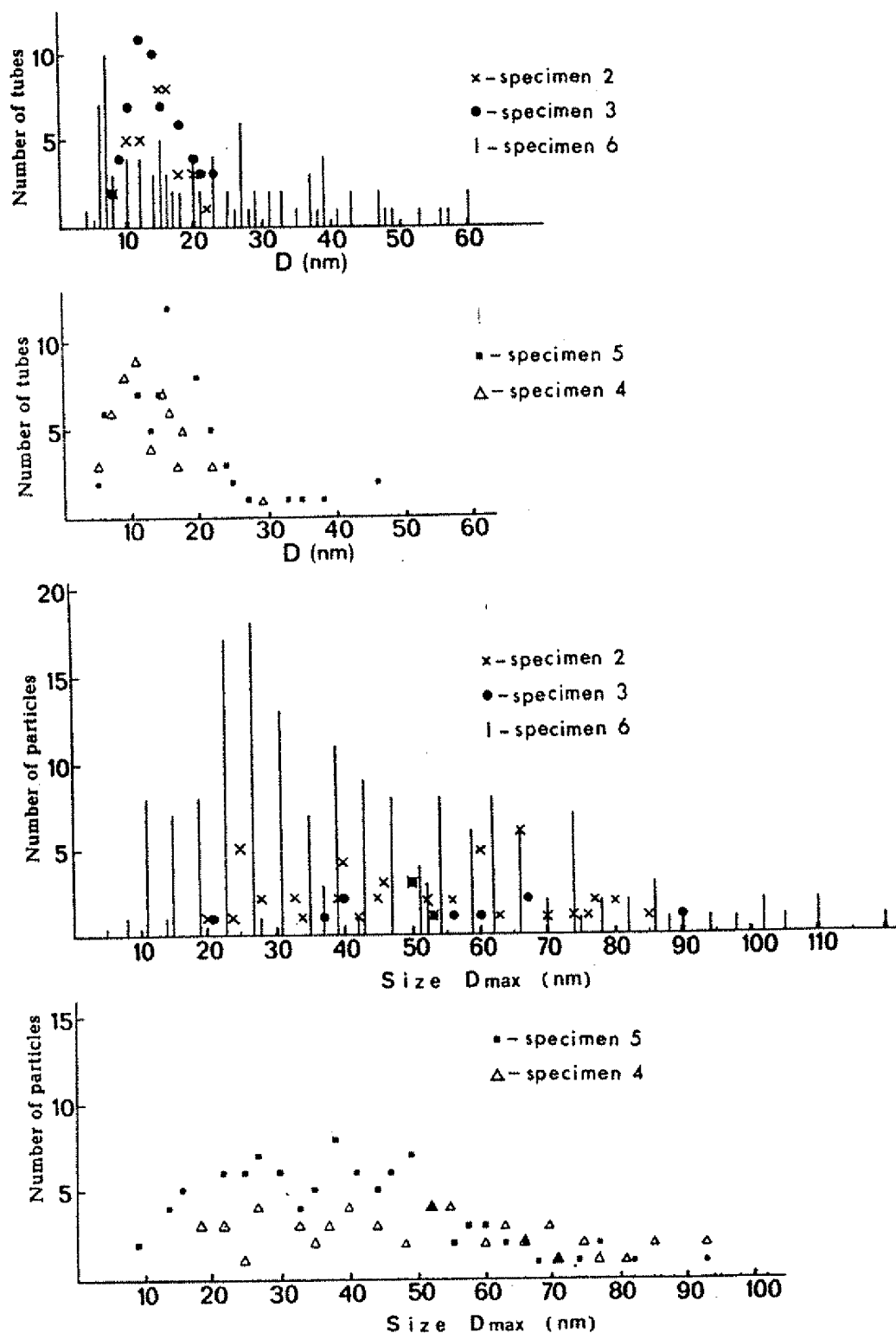[a] Pressure values in the reaction chamber during the arc operation.

Fig. 3. Distributions of nanotube diameters (a, b) and linear sizes of isometric multilayer polyhedral particles (c, d). (Numbers of measured MWNs and MPPs for histograms are shown in parentheses).

MPPs (Fig. 4). The range of nanotube diameters (Fig. 3a) was much wider than that for specimens 1, 2 and 3. So were the sizes of the MPPs (Fig. 3c). Again we observed a much wider range compared to 1, 2 and 3 samples. Thus

higher He pressures facilitate broadening of the size distributions of both the tubes and the MPPs.

The influence of the feed velocity on the composition of the deposit is exemplified by entries 4 and 5 in Table 1. At
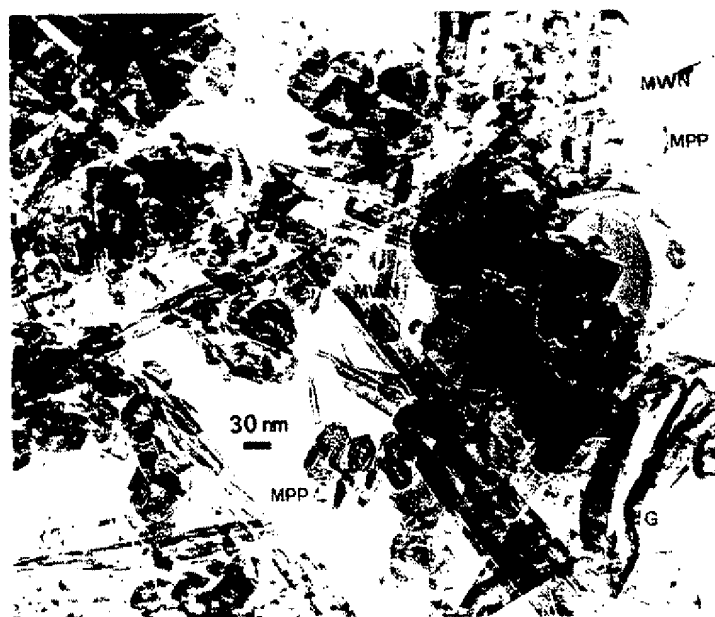
Fig. 4. TEM image (250 keV) of specimen 6. The main components are multilayer polyhedral particles (MPPs), nanotubes (MWN) and graphite formations (G).

a feed velocity equal to 2.5 mm/min (specimen 5) we were close to its optimal value at 500 torr He pressure and 65 A arc current for 6-mm diameter graphite rods. It should be noted that at a low feed velocity (specimens 5 and 6) a wider range of nanotube diameters is prominent. Nanotubes of very small diameters, very small-sized MPPs, as well as very thick MWNs were observed. Thus lower feed velocities (wider gaps) facilitate the broadening of size distributions in a manner similar to increasing the He pressure.

It should be stressed that no sole parameter or its optimal value may be identified at present as the most crucial factor for determining the deposit's components. For example, comparable yields of MWNs can be obtained with either a narrow or wide gap-width. Actually the composition of a deposit will be dependent mainly on the temperature and carbon vapor properties inside the gap, which are determined by a multitude of factors, including He pressure, carbon arc power, graphite rod size etc. (see [14]). Therefore, at present the combined influence of all the parameters must be considered in the search for the optimal conditions of MWNs synthesis.

It would be of interest to characterize the lengths of nanotubes using histograms. This task still remains mainly because of the aggregation of the tubes. Measurements of lengths of various nanotubes encountered in deposits (though not the distributions because of tangled nanotube aggregations) have been performed with SEM.

On the basis of TEM measurements it has been possible to select nanotube rich specimens. These are represented in Table 1 by specimens 6, 7 and 8. Besides MWNs they contain MPPs and graphite. 3D organization of these components in the deposit core is also of interest. But it

cannot be revealed by TEM because the sample preparation completely destroys the deposit structure. Only secondary aggregation of components will be exposed by TEM and HREM, like nanotube side-by-side bundles and large agglomerates of MPPs. SEM was the better instrument for the examination of the internal deposit structure, the localization of the main deposit components, and the exploration of the orientation of the nanotubes.

3.2. SEM investigation of the internal organization of deposits

In this section the 3D organization of the deposits is considered as revealed by SEM measurements. Nanotubes are better distinguished by the SEM compared to MPPs and similar formations, which are liable to aggregation. MPPs in SEM images were identified as the isometric particles that were ~30–100 nm in size.

In accordance with earlier reported results [4–6,9,10], the soft black core of a deposit obtained under favorable conditions for the production of nanotubes was composed of parallel columns of about 50–60 μm in diameter aligned along the axis of the deposit growth. The top growth surface of the deposit was almost flat when the optically black core was most abundant. This case corresponded to high helium pressures and low arc currents, which were preferable for a higher yield of nanotubes. An SEM top-view image of a region of the growth surface of a deposit (Fig. 5a) shows the tops of these columns forming a honeycomb-like structure. Viewing the surface at an angle of 70° to the deposit axis reveals that the tops of the columns are nearly hemispherical (Fig. 5b). The tops of adjacent hemispheres were separated by 65–75 μm, that is
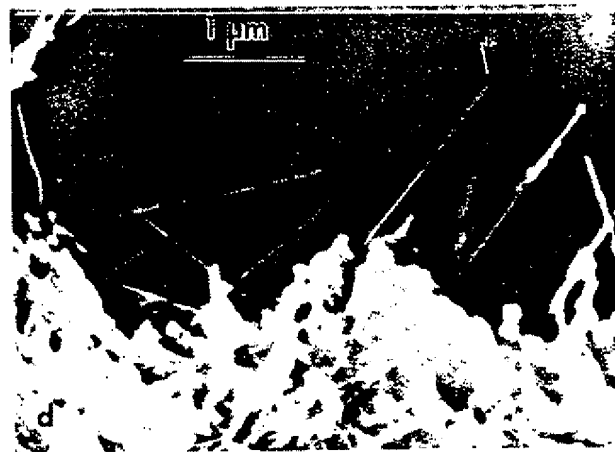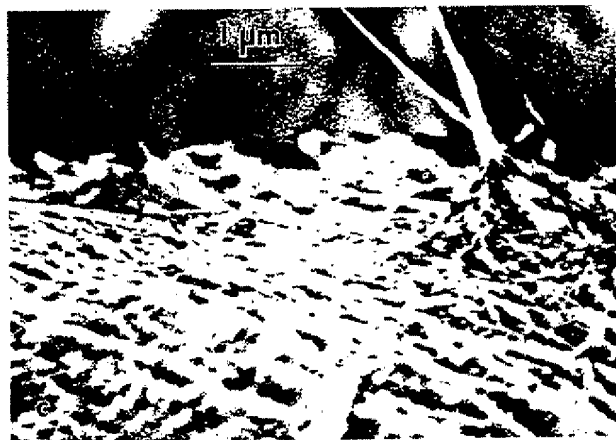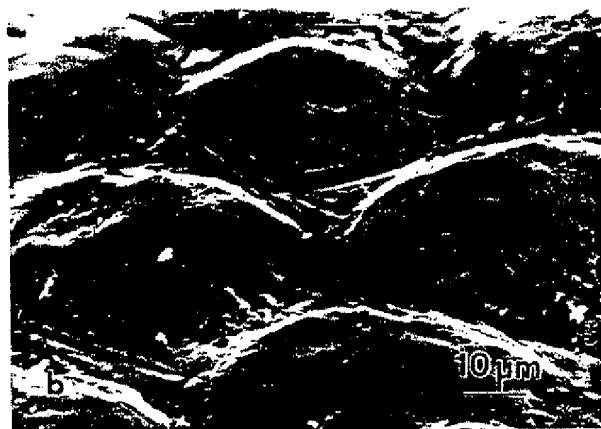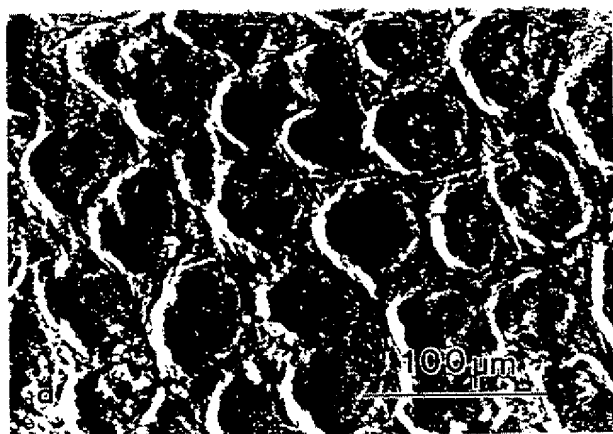
Fig. 5. SEM images of the growth surfaces of typical deposits. (a) A top view at low magnification of sample 7; (b) hemispheres at the column ends at higher magnification (side view taken at 70° to the deposit axis) of sample 7; (c) a separate hemisphere covered with nanotubes and their bundles that lie on the growth surface (side view taken at 60° to the deposit axis); sample 8 (it is characterized by higher (11.3 mm) deposit diameter then other samples, but shows similar column structure); (d) side view of the surface of a hemisphere at high magnification (taken tangential to the surface); nanotubes down to 20–30 nm in diameter and up to 4 μm long (arrowed) are clearly seen; sample 6.

substantially less than the corresponding distance reported in Ref. [6], probably because of the difference in arcing conditions. A closer examination of a separate hemisphere on the growth surface of a deposit just removed from the arc apparatus (that avoided the accidental removal of the delicate nanotubes from the surface) revealed a layer of fibers densely covering the top of the column (Fig. 5c). These fibers according to their dimensions and morphology could be like the nanotubes or their bundles seen in our TEM and HREM investigations. It is likely that such a covering layer had been the source of the MWNs discovered with the HREM by Iijima [8]. Most of the nanotubes that covered the tops of the columns lay along its upper surface or at angles exceeding 45° from the axis. In some places tubes extended from the covering layer (Fig. 5d), showing their diameters to be equal to 20–30 nm. That was consistent with the TEM data for the separate nanotubes. These protruding nanotubes led to the possi-

bility of using separate columns several mm long as electron field emitters.

The typical cross-sectional view of a deposit also reveals the friable columnar structure of quasi-hexagonal ordering mentioned above, with distances between the column axes of about 65 μm (Fig. 6a). As seen in Fig. 6a, the appearance of the cross-sections is usually step-like with transverse and longitudinal (with respect to the deposit axis) areas. After sectioning, longitudinal areas of steps (up to ~1 mm long) always pass between columns. Some protruding columns nearest to the step edges separate at their ends making the observation of their lateral sides convenient at various angles. Sections of columns themselves were usually normal to their axes or slightly inclined to them. At a higher magnification (Fig. 6b) it is clearly seen that columns 60 μm in diameter in a given fragment are separated by a low density intercolumnar space of 10–15 μm as distinct from about 50 μm
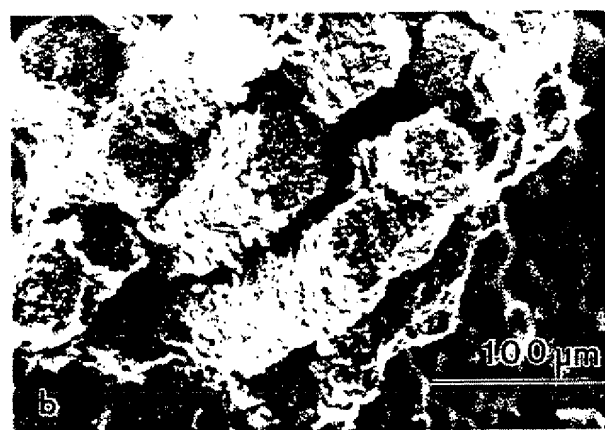
Fig. 6. SEM image of the cross-sectioned deposit (specimen 7). The growth axis is directed from the observer along the columns. (a) The columnar structure at low magnification. Friable columnar structure and dense layered structure (upper right) are seen there. Aggregates of columns with cross-sectional dimensions of about 0.5–1 mm attract attention. The arrowed area is shown in (b) at higher magnification. (b) At higher magnification the most friable area between the columns is well discernible. After splitting, some columns have separated.
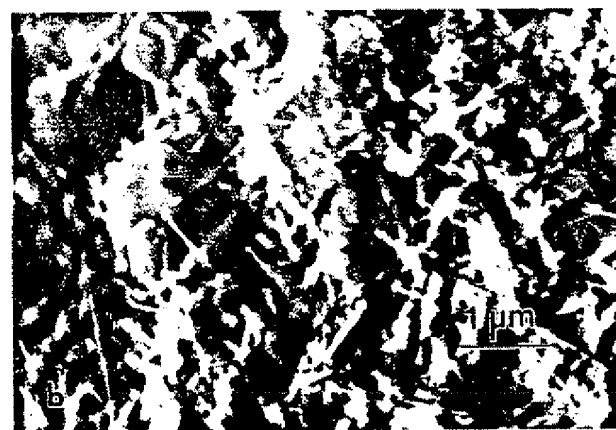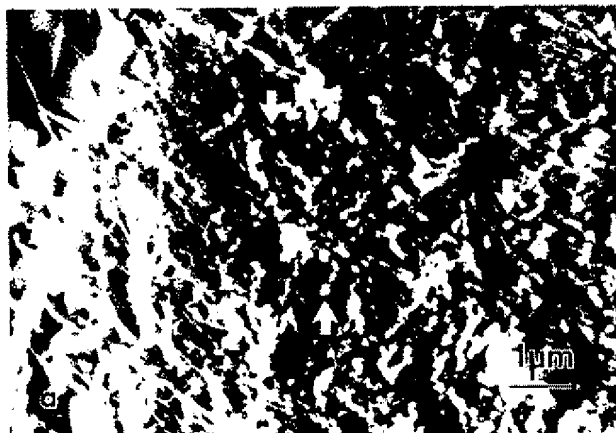
Fig. 7. SEM images of the cross-sectional view of the sectioned separate column (specimen 7): (a) near its edge (the growth axis is directed upwards and is inclined at the top of the image ~20° from the image plane). One can see the lateral braiding of nanotubes (the bright nanotube area on the left characterized by high secondary electron emission) and the internal section of the column (on the right). Inside the column there are no nanotube co-oriented structures having uniform alignment, including any structures along the column. Some spear-like tubes are directed outwards from the lateral side (the braid) of the column; (b) near the center of the column: normal view: the growth axis is directed from the observer. Particles that can be considered as MPPs are indicated with arrows.

intercolumnar spacing observed earlier [6] after the somewhat lower arc-current deposition densities. Our investigation shows that the highest concentration of nanotubes is attained in those low density intercolumn spaces and at the side surfaces of the columns. SEM examination of a cross-sectioned separate column (chosen from the cross-sectional deposit view) reveals an area of enhanced emission of secondary electrons along its circumference (Fig. 7a). This area is composed of interlaced nanotubes forming an outer braid to the column. The characteristic thickness of this (measured on protruding columns) was about several micrometres. The outer diameter of protruding columns varies along their length in a somewhat periodical manner (Fig. 6b). The inner part of a column inside the braid (to the right of the braid in Fig. 7a) consists of a disordered mesh of various forms of graphite mixed with 50–70 nm diameter MPPs and smaller amounts of nanotubes. If there had been any longitudinally

such an observation. Randomly oriented nanotubes amidst other particles were also revealed by the SEM (Fig. 7b). No tubes aligned along the deposit axis were seen in Fig. 7b and the like (they would have been observed as a collection of very bright dots). It can thus be stated that in the columnar structure of the black core, the MPPs and curved graphite particles were concentrated inside the columns. It should be noted that MPPs and graphite components were rather tightly embedded inside the columns and often overlapped each other, leaving no room for longitudinal void channels, where co-oriented long nanotubes or their bundles could be formed. No preferential co-orientation of nanotubes or their bundles was seen inside cross-sectioned columns, despite observations at
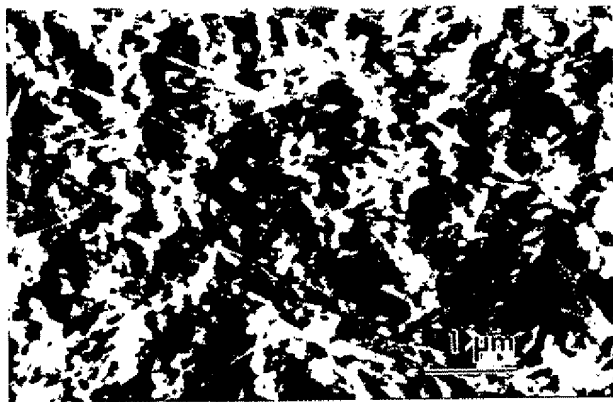
Fig. 8. Typical SEM image of the lateral side of a separate column revealing the nanotube braid and its structure (specimen 7). The direction of observation is normal to the lateral column side. Groups of tubes oriented at ~70° to the growth axis (the vertical line) are clearly visible. Few tubes are oriented close to the vertical direction, there are practically no tubes oriented perpendicularly to the growth axis.

Typical SEM image of the side surface of an intact column taken in the direction normal to column axis (Fig. 8) shows the structural organization of the outer layer of the braid. It consisted predominantly of interlaced nanotubes and their bundles. The thinnest observed tubes were 20–30 nm in diameter, that agreed with the TEM data. No significant inclusions of components other than nanotubes were observed in these outer layers. It is important to estimate the average direction of nanotubes in the braid with respect to the deposit growth axis. Analysis of column side views (about 25 μm^2 in size) has shown that in most of them, more than 50% of the nanotubes or their bundles are inclined in the micrograph plane at angles exceeding 45° relative to the deposit axis. Side images (like that shown in Fig. 8) often reveal that noticeable part
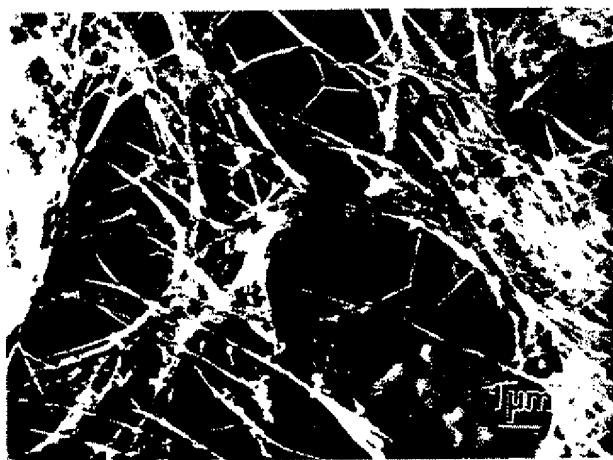

Fig. 9. SEM image of the nest-like structure of nanotube bundles found in the interspace between columns in the oxidized specimen 7.

that correspond to 45–75 relative to the axes. Many of them are tangential to columns. Few tubes lie along axes or normally to them. Thus we can conclude that nanotubes in the braid covering the columns are inclined on average more than 45° with respect to the axis of a deposit.

Oxidative etching of columnar structures in air helps to reveal (or produce) fairly pure arrays of nanotubes located in the intercolumnar space. They often form nest-like structures (Fig. 9). The thinnest nanotubes observed in the intercolumnar space (~20 nm in diameter) were protruding from the braid as spear-like tubes seen in Fig. 5d.

Though in the majority of columnar structures there was a very noticeable content of nanotubes, on the basis of our examinations it should be noted that the existence of columns in deposits does not necessarily bear evidence about high concentration of nanotubes between columns and inside them. In some deposits, the columns had a low content of nanotubes both at their peripheries and inside them. Such columns may consist predominantly of other types of carbon particles, e.g. of variously stacked graphene layers. Growth surfaces of such columnar structures are not as regular as in the case of nanotube rich deposits. Thus, the formation of columnar-like structures in deposits is a necessary condition, but not the only condition for the production of high concentration of nanotubes in deposits.

The elements of the described columnar structures are rather fractal-like. This comes out of the fact that similar SEM images are often registered at different scales. That means that small and large details of the structures are geometrically close to each other except for scale. Thus on a cross-section of a deposit at essentially low magnifications one can see (Fig. 6a) clear agglomerates of similar columns ~1 mm in diameter that resemble separate columns, and at greater magnifications inside these columns one can find spatial structural formations of composites ≤10 μm in diameter surrounded by nanotubes. This demonstrates the *self-similarity* [15] of the columnar 3D structures, which is the important property of fractals. The honeycomb-like column structure described above has much in common (see also Ref. [6]) with the Bénard convection cells in fluids that are fractals [16]. So nanotubes grow as an essential part of a fractal-like solid that cannot be synthesized in the linear system, but only in a complex non-linear dynamical system [15,16], which in our case is the arc plasma. This result is important for the search for better technology of nanotubes production in deposits.

4. Discussion

The reported results are in accordance with earlier electron microscopic observations of cathodic deposit structures [4–6] except the issue of the nanotubes location

and orientation in a column, denoted in Ref. [6] as zone 1. The columns are generally thought to be composed of MWNs aligned along their axes as distinct from the picture outlined above: nanotube braided columns filled with randomly oriented nanotubes, MPPs and graphite. There are several plausible reasons, which might account for the discrepancy. First is the different sample preparation procedure for SEM observations, which remains poorly defined in other papers. For example, the use of a microtome can quite easily cause the alignment of tubes along the direction of the cut, as has been the result of slicing the composite polymer resin matrix containing originally randomly dispersed nanotubes [11]. The other reason may be the intrinsic uncertainty in the actual orientation of a very thin object that is used for HREM observations, again due to the method of sample preparation for viewing a tube down its axis [5,12]. Finally, the difference in the arcing conditions can cause drastic variations in the columnar structure. Our experience rejects the latter, for we have not observed the axial orientation of nanotubes in columns in any of the numerous samples studied. Nevertheless, further SEM studies of different columns with the highest possible resolution seem pertinent.

An indirect indication of the direction of nanotube orientation in columns can be derived from deposit magnetization anisotropy measurements, which reveal that the value of longitudinal diamagnetic susceptibility for deposits is about 10% higher than the value of the transverse susceptibility [5]. Under the assumption that the longitudinal component of susceptibility dominates in a tube, the conclusion has been made that tubes must be oriented preferably along the deposit axis to explain this observed macroscopic anisotropy [5]. Quite recently it was experimentally substantiated that this assumption was incorrect [13] and that the nanotubes in the deposit investigated in Ref. [13] were preferably oriented at high angles to the deposit axis, in complete accordance with the results of the present work achieved from direct SEM observations of similar deposit samples.

The structure of the nanotube rich columns established in the present work does not correspond to the hypothesis about the alignment of tubes along the column axis, which is an essential point in a model [6] offered to describe the growth of a deposit in a direct current arc. In this model the tips of parallel nanotubes in zone 1 (top of columns) act as field emitters of electrons into the plasma. This electron injection produces a high degree of carbon ionization resulting in a carbon ion current flow concentration above columns, that provide the principal feedstock for their growth. The helium buffer gas is drawn in by the carbon ion flux to the tops of the columns and then sweeps to their sides and returns back to the plasma over zone 2, the intermediate area between adjacent columns.

Some refinement of this model seems necessary in view of data on the column structure discovered. The emission

of electrons from tops of columns (zone 1) in the absence of a large amount of vertically oriented nanotubes (Fig. 5c and d) may be thought to occur as mainly thermionic in its nature from all the constituents of columns, at the same time keeping the possibility for the occurrence of the field emission from inclined nanotube tips and sharp edges of other particles, either formed at the surface or even fallen down (as we suppose) from the plasma volume. The dominating abundance of horizontally packed nanotubes over the column top surface (Fig. 5c) probably indicates that the electric field effect at the open end of a nanotube [6,17] is not a governing factor for its growth. Those nanotubes (Fig. 5c) that were originally located near the very hot top of a hemisphere could then undergo evaporation, yielding neutral carbon particles. An estimate has shown [6] that the majority of carbon precipitated on columns must evaporate back as neutrals. This evaporation enriches the central part of a column in nanoparticles and graphite to the extent finally observable with the SEM (Fig. 6b), as these components are thermodynamically more stable compared to nanotubes [17]. At the periphery of a column the evaporation is less effective and nanotubes mostly survive, thereby forming the braid of a column (Fig. 7a and Fig. 8). Obviously nonreactive helium carries re-evaporated carbon neutrals to zone 2, where they serve as a principal feedstock for the growth of new nanotubes [6].

Because of the existence of columnar structures with a low nanotube concentration it is evident that the presence of nanotubes may not be vital for column formation in the deposits. It can be supposed that nanotubes grow in preference on columns just because of favorable conditions for their growth there, for example a suitable temperature range at the growth surfaces. Nevertheless, when formed nanotubes probably stimulate more regular and stable column formation, since nanotube rich deposits generally have a more explicit and regular columnar structure, as compared to cases of low nanotube concentration. The reciprocal influence of columns and nanotubes needs further exploration.

As has been found, nanotubes were most abundant in the intercolumnar space and in the braids of columns (Fig. 7a, Figs. 8 and 9), and this may serve as a hint for developing the technique for their separation and purification. Besides this, the structures of zone 1 and zone 2 disclosed in this work give an explanation for the generally irreproducible results of repeated TEM analysis of the composition of differently prepared samples of the core material.

From the conclusion (based on the fractal character of columnar structures) that columnar deposit formation and nanotube growth take place in a nonlinear process in the arc plasma, several important things for the deposit growth come out: (1) almost all the parameters of the arcing process are interdependent: an attempt to adjust only one of them will lead to inevitable variation of others; (2) no sole parameter or its value can be regarded as the most

nological result (for example high nanotube yield) could be achieved; (3) several sets of controllable parameters can give similar results; (4) not all the sets of experimental parameters provide stable growth. Sometimes oscillations of growth processes can take place and periodic changes of the structure in the deposits can be observed: for example, along the deposit axis loose columnar structures can be periodically displaced by dense sliced structures and vice versa. As it is not productive to stabilize growth processes in unstable domains of the growth, it is of prime importance to find these islands of stability in the ocean of possible experimental parameters for appropriate deposit production [5–12,18]. The conditions listed in Table 1 illustrate important solutions in our search for better production of nanotubes.

5. Conclusions

With the help of a thorough electron microscopical investigation, using both TEM and SEM techniques, we have clarified the main peculiarities of the columnar structures of the nanotube-rich deposits prepared under stable arcing conditions. Taking into consideration these newly observed peculiarities (the absence of co-oriented longitudinal nanotubes or their bundles and the preferential orientation of nanotubes in column coverings at high angles to the deposit axis) the refined stationary model of deposit growth has been outlined, that is in accordance with our observations by electron microscopy. These peculiarities might be helpful in the explanation of existing confusion that arises because of the effects of deposit preparation and further sample handling.

Acknowledgements

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EXHIBIT 19

High Performance Fibres from 'Dog Bone' Carbon Nanotubes**

By *Marcelo Motta, Anna Moisala, Ian A. Kinloch, and Alan H. Windle**

Single- and double-wall carbon nanotubes (SWCNTs and DWCNTs) are promising candidates to serve as building blocks for a new generation of high-performance fibres. Both theoretical models and experimental data show that the axial strength and stiffness of individual SWNTs are of the order of 50 GPa and 1 TPa, respectively.[1-4] However, their assembly into useful forms, such as macroscopic fibres, with properties which reflect a significant proportion of those seen in the individual tubes, remains a well-defined challenge to materials processing.

We have recently introduced a method for the direct spinning of pure carbon nanotube fibres from an aerogel formed during chemical vapour deposition (Fig. S1).[5] The continuous withdrawal of product from the gas phase as a fibre (Fig. S2) imparts high commercial potential to the process, including the possibility of in-line post-spin treatments for further product optimisation. We have more recently shown that the mechanical properties of the fibres are directly related to the type of nanotubes present (i.e., multiwall or single wall), which in turn, can be controlled by the careful adjustment of process parameters.[6]

Fibres based on SWCNTs alone have been successfully produced by conventional spinning from lyotropic solutions in super acids. Such fibres have a relatively high modulus (130 GPa) but moderate strength (0.2 GPa), despite the well-aligned microstructure.[7] Fibres spun from substrate-based 'forests' of MWCNTs possess a tensile strength of 0.5 GPa (~ 0.6 N/tex) which is attributed in part to the degree of twist applied during spinning.[8] Polyvinyl alcohol (PVA)-SWCNT (~ 60 wt%) composite fibres with a tensile strength of 1.8 GPa (1.2 N/tex) and remarkable toughness (570 J g^{-1}), albeit at a strain to failure of around 100%, in samples which were pre-strained by about 200%, have been reported by Dalton et al.[9] In a more recent publication based on essentially the same system (50 wt % nanotubes), Miaudet et al.[10] have obtained a strength of 0.55 GPa (~ 0.4 N/tex) and a re-

markable energy at failure of 870 J g^{-1}, although in this case the maximum strain to failure observed was 430%. The authors point out that for many applications, such as body armour, it is the energy absorbed at low strains that is significant and that their fibre had absorbed around 10 J g^{-1} at 10% strain. By hot-drawing PVA-SWCNT composite fibres, the authors report an increased strength of 1.6 GPa (~ 1.2 N/tex), while the fracture strain was reduced to 11% and the toughness at this strain was an encouraging 85 J g^{-1}.[10] For direct-spun CNT fibres,[5] we have previously reported 0.70 N/tex strength and 40 J g^{-1} toughness.[6] In this paper we describe improvements in these values and relate the performance to unique aspects of fibre microstructure. In particular we have developed successful strategies to avoid particulate defects amongst the network of nanotube bundles forming the fibres.

Recent improvements in the process have enabled us to spin continuously with iron contents down to 25 ppm. At present, these fibres are spun at a rate of ~ 5–25 m min^{-1}, with diameters in the range 2–20 μm and linear densities ranging from 0.05 to 0.5 g km^{-1} (tex). The density of the fibre depends on the degree of condensation of the carbon nanotubes, which is a function of post spinning processing such as twisting and/ or the wetting and evaporation of volatile organic liquids such as acetone. Fibres, such as that shown in Figure 1a have a density of around 1 g cm^{-3}. However, the fibre thickness is defined in terms of units of tex, the weight in grams per kilometre length, a parameter which does not require measurement of either diameter or density, both of which are difficult to acquire accurately for fine fibres.

At the resolution of the scanning electron microscope (Fig. 1b) the fibre structure consists of an oriented network of bundles, some that divide and rejoin, typical of the organisation of single-wall nanotubes (individual thin-walled nanotubes (single or double wall) are below the resolution limit of this technique). Figure 1c is a high resolution TEM image of a typical bundle, the diameters of which are in the 20–60 nm range. While there is no evidence of multiwall tubes in any of the samples, it is very difficult to identify particular single- or double-wall tubes within the bundles. The groups of parallel lines which can be seen can be followed over considerable distances (several micrometers) so there is no evidence of nanotube ends either. However, where discrete tubes separated from bundles were seen, as shown in Figure 1d, they were of surprisingly large diameter (in the range of 5–10 nm) and mainly double-wall.

A key indication to the internal structure of the branched bundles forming the continuous network came from the examination of the bundles close to a fibre fracture. Figure 2

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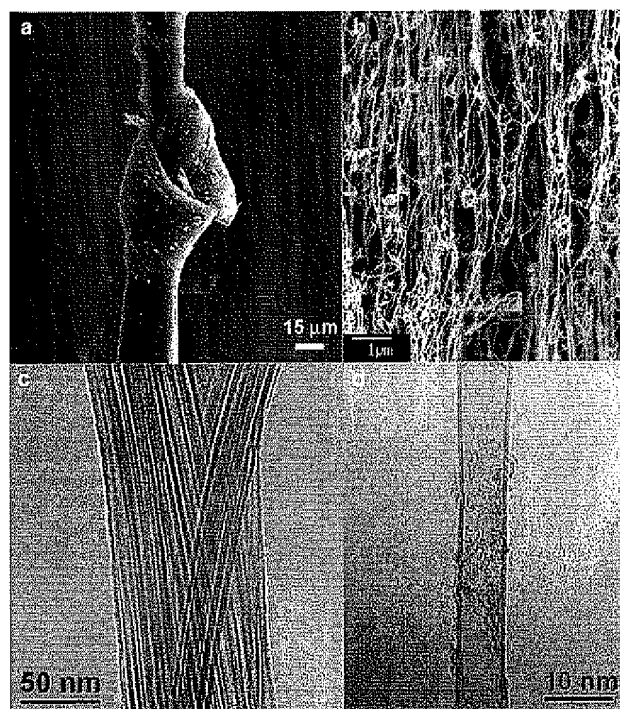E-mail: ahw1@cam.ac.uk

Figure 1. Structure of the fibre product. a) SEM image of a knotted fibre (0.4 tex) stressed to 0.5 N/tex. b) SEM image showing the microstructure of the fibre at a higher magnification. The bundles form a continuous network as they branch and rejoin. They are predominantly oriented along the fibre axis (vertical). This fibre had not been condensed by post treatment to the same degree as that of Figure 1a although it had the same tex, as the continuous network is more easily visible in the slightly more open structure. c) High resolution HRTEM image of a bundle. This particular one shows the beginning of the division associated with the network points typical of SEM micrographs of single- or double-wall material as in (b). While the striations along the bundle are suggestive of it consisting of overlapping thin walled nanotubes, it is difficult to distinguish them. d) HRTEM of a discrete double-wall tube of diameter 7.4 nm broken away from one of the bundles such as that in (c).
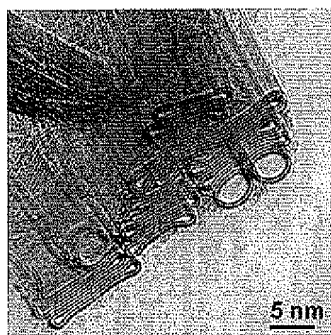

Figure 2. HRTEM image of a bundle close to a fibre fracture revealing that the bundles consist, predominantly, of collapsed double-wall nanotubes greater than 5 nm. Note both the "dog-bone" cross section of these tubes and the non-collapsed tubes at the edge of the bundle. Also present is a collapsed triple-wall tube (14 nm equivalent diameter) in the centre of the image and a few collapsed single-wall tubes greater than 4.5 nm. An analysis of the tubes forming this bundle is in Table 1.

shows a fractured bundle and is most revealing. It is clear that the tubes are mainly double-wall, although there is one triple-wall and five single-wall. Furthermore, all those with a circumference corresponding to diameters larger than ~ 5 nm have collapsed, generating what are, in effect, stacks of parallel graphene layers. Table 1 is an analysis of the tubes in this particular bundle. Other separated bundles have been observed and they show the same structure. One such is shown in Figure S3.

Table 1. Analysis of the nanotube bundle shown in Figure 2.

Equivalent diameter [nm]	No. of walls	Collapsed (Y/N)	Equivalent diameter [nm]	No. of walls	Collapsed (Y/N)
4.6	1	Y	6.5	2	Y
4.7	1	Y	6.8	2	Y
4.8	1	Y	6.8	2	Y
5.2	1	Y	7.9	2	Y
5.7	1	Y	8.3	2	Y
4.2	2	N	8.3	2	Y
4.6	2	N	8.4	2	Y
4.7	2	N	14	3	Y
6.2	2	Y			

The observation of collapsed nanotubes is the clearest experimental evidence to support the modelling prediction of Elliott et al.[11] of auto-collapse of single-wall tubes under atmospheric pressure when the diameter is greater than a critical value in the range of 4.2–6.9 nm (see also Kim et al.[12]). For double-wall tubes it is considered that the critical diameter will be fairly similar, when it is the inner component which is measured.[13] The extra strain energy associated with the higher curvature at the "dog-bone" ends of the collapsed tubes is more than compensated for by the energy of bonding between the inner surfaces of the graphene sheets. The previous modelling work also indicates that the critical collapse diameter of a double-wall tube would be greater than for single-wall tubes, as it is the diameter of the inner component. In this context note from Table 1 that while the single-wall tubes observed, having diameters in the range of 4.59–5.66 nm, had all collapsed, the three smallest double-wall tubes with equivalent diameters in the range 4.24–4.73 nm, had not collapsed.

Figure 3a is a typical electron diffraction pattern from a single bundle showing very clear 002 maxima and some preferred orientation of the $hk0$ rings. There is also significant streaking of the 002 maxima towards the centre of the pattern (the first sharp ring is an artefact of the microscope). Figure 3b is a powder X-ray diffraction (XRD) scan of a significant volume (~1 mm³) of unaligned fibres. The 002 peak is clear, as is a broad halo of intensity stretching from it towards lower angles. It is consistent with the equatorial streak on the electron diffraction pattern. The position of 002 is at 26.3° 2θ (Cu Kα) corresponding to 0.340 nm, which is typical of multiwall CNTs and is very close to the turbostratic graphene layer spacing of 0.344 nm.[14]
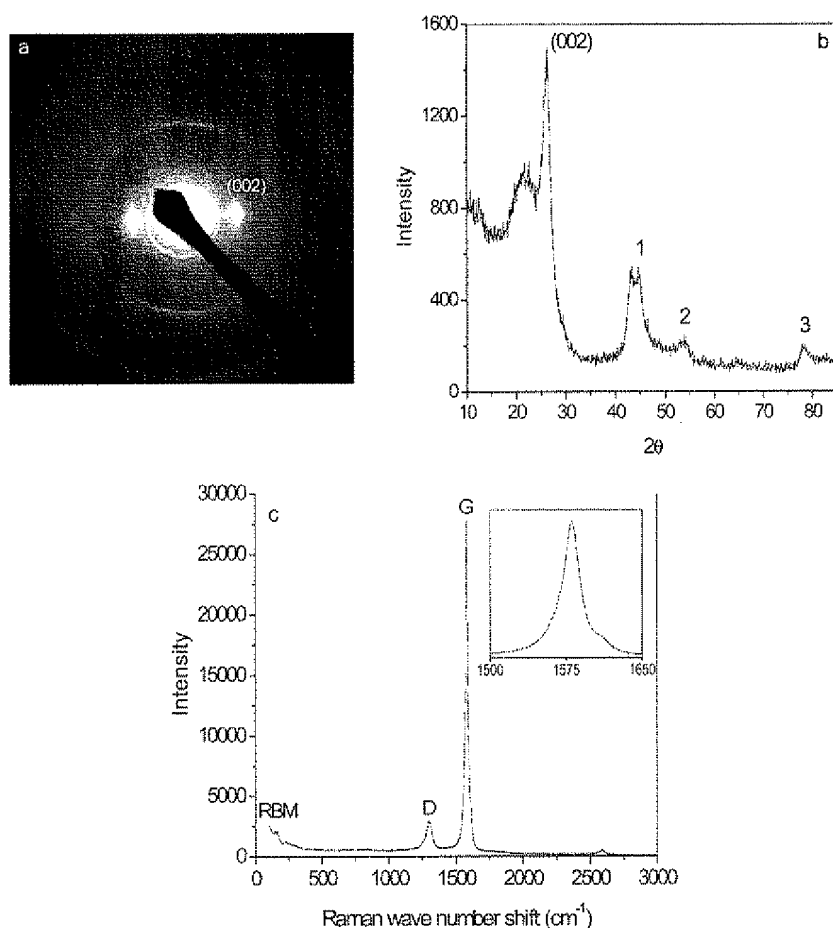
Figure 3. a) Electron diffraction pattern of a single bundle of tubes. The bundle axis is vertical with respect to the page. The marked equatorial reflections are 002 indicating the quality of orientation of nanotubes within the bundle. The intensity streak along the equator at lower angles than 002 indicates that some of the interlayer spacings are greater than that typical of graphene stacks. The continuous bright ring at low angles is an instrument artefact. b) Wide angle X-Ray diffraction pattern, using reflection geometry and Cu $K\alpha$ radiation, from an orientationally randomised sample of fibres. Again there is significant diffuse intensity at angles below the 002 peak. Peak 1 is a combination of carbon 100 and 101, and catalyst residue peaks (in fact a mixture of FCC iron 111 and BCC iron 110) while peaks 2 and 3 are also catalyst residue peaks corresponding to FCC iron (200) and (220), respectively. c) The Raman spectrum with a small I_D/I_G ratio (0.1) is an indication of well-ordered nanotubes. The inset shows a splitting on the high wave number side of the G band, which is typical of multiwall tubes. The spectrum also shows low intensity RBM peaks. The radiation wavelength was 633 nm, although similar RBMs were found with 514, 785, and 830 nm lasers.

further characteristic of single-wall nanotubes. On the contrary, the spectra show a typical MWNT feature, i.e., a subsidiary peak on the high wave number side of the G band. The G band is, nevertheless, sharp and much more intense than the D peak, indicating well-ordered nanotubes. Another aspect in which the Raman spectrum is 'non-typical' is that the G' peak at 2590 cm^{-1} is very low in intensity compared with spectra typical of multi- or single-wall carbon nanotubes. In some respects this weak peak echoes the peak seen from small numbers of superimposed graphene layers, and just beginning to be discussed informally.

Viewed in isolation, the diffraction and Raman data are strongly suggestive of multiwall tubes. There are sharp 002 diffraction maxima, albeit heavily 'streaked' to lower angles, which are much sharper than would be expected from double (or even triple) layers. Furthermore, our direct-spun fibre shows a G band form characteristic of MWNTs, there being a small subsidiary peak at very slightly higher wave numbers. There is some evidence of RBM peaks, but these are comparatively weak. Yet, no multiwall tubes were seen in any of the microscopy.

However, the presence of discrete 002 peaks is exactly what is to be expected from stacked layers of collapsed nanotubes. The broad smearing of the 002 peak towards lower angles correlates with the regions of the collapsed structure where the walls of adjacent nanotubes are not quite in contact. Furthermore, the compatibility of the diffraction evidence, both X-ray and electron, with the collapsed nanotube structure seen by microscopy underlines that the collapse of the tubes, was not the result of the fracture process, nor caused by sample preparation for microscopy but is an intrinsic feature of these direct-spun carbon nanotube fibres.

The radial breathing mode (RBM) peaks seen at low wave numbers in Raman spectra are taken as the fingerprint for single-wall nanotubes. The spectrum from direct-spun CNT fibres (Fig. 3c) showed peaks in the 128 to 245 cm^{-1} wave number region, which would correspond to single-wall nanotube diameters in the range of 1 to 2 nm.[15] However, the intensities and definition of these peaks are significantly lower than is expected for fibres consisting predominantly of single-wall nanotubes, and probably represent a very small volume fraction of such tubes. Additionally, the G band lacks the subsidiary peak on its low wave number side (e.g., 1570 cm^{-1} for a single-wall nanotube of 1.4 nm diameter), which is again a

Another consequence of such a structure is that any detailed interpretation of the low wave number end of the Raman spectra would be wrought with difficulty. It should also be noted however, that observations of single-wall nanotubes collapsed by high hydrostatic pressure also show a marked broadening and loss of definition of the RBMs.[16] The MWNT indications from the form of the G band may also be associated with stacking of the collapsed tubes within the bundles.

Polarised Raman studies of these direct-spun fibres show a minimum G band intensity at 90° in respect to the fibre axis. The intensity is ~ 17 % of the maximum intensity observed in parallel configuration, implying a high degree of tube orientation along the fibre axis.[17] Qualitatively, the high degree of nanotube alignment indicated by polarised Raman measurements is in line with the microscopic observations of the fibre structure. The quantification of these measurements for our particular structure also requires further basic studies. Both theory and experiments show polarisation dependence of the D and G bands of carbon nanotubes with the maximum signal intensity observed when light is polarised along the tube axis. With individual single-wall tubes the G band signal is fully suppressed at 90° due to the depolarisation effect.[17] Neighbouring tubes affect the depolarisation process, yielding non-zero intensities. For multiwall tubes, especially as the diameter increases, the effect is less pronounced and the minimum (non-zero) intensity would be observed at a polarisation angle of ~ 55°.[18] This issue must remain open in the context of collapsed double wall nanotubes.

Overall, we observe three hierarchies of structure within the fibre. At the fundamental level, there are the nanotubes which are mainly double-wall in the diameter range of 5–10 nm. At the next level the nanotubes have aggregated into bundles of 20–60 nm in diameter, and the nanotubes are collapsed into dog-bone cross sections which then produce stacks of parallel graphene sheets. At the microstructural level these bundles form a continuous network with preferred orientation along the fibre axis.

Figure 4a shows stress-strain curves from the fibres made under the standard conditions defined in the experimental section. The tex of the fibre produced was of the order of

0.4 g km^{-1}. The curves are interesting and show some of the features seen in earlier examples from less than optimum processing conditions. The strengths are a little above 1 N/tex and the stiffness some 60 N/tex. It is significant that the curves show a yield stress followed by a region of plastic deformation prior to fracture at strains in the 4–8 % range.

A consequence of the plastic deformation is that the energy absorbed at fracture lies between 30 and 60 J g^{-1}, which is competitive with the best high performance fibres available. In spite of the potential role of such fibre in applications where energy absorption is of the essence, the toughness also imparts much higher degrees of damage tolerance in applications requiring weaving or clamping of the fibre. One test of such tolerance is the knot strength, in which the strength of a knotted sample (simple knot) is compared with the unknotted fibre and the result expressed as a percentage. Figure 1a shows such a test. For our CNT fibres with strengths of the order of 1 N/tex, the knot strength is 70 %, while one or two examples actually broke in sections remote from the knot. In comparison, the knot strength of carbon fibres (e.g., Thornel 300) are in the region of 5 %, while aramids (e.g., Kevlar 49) are of the order of 35 %.

In a study of the nature of the aerogel of nanotubes formed in the hot zone of the CVD furnace, the emerging thread was wound out of the tube at increasing speeds until the reaction could no longer keep up so the aerogel was pulled right out of the furnace breaking the continuous spin. The fibre produced under the maximum wind-up speed at which continuity can be maintained, 25 m min^{-1}, had a lower tex (~ 0.2 g km^{-1}) but enhanced strength.

Figure 4b shows three tests on samples of such a fibre with strengths in the region of 2 N/tex. It appears that these values result from drawing of the aerogel in the hot zone as the nanotubes are actually being formed, the tex of the fibre collected being reduced from 0.4 to 0.15 as a result. The three curves shown are selected from the centre of a wider distribution of strengths. The weakest fibre seen was 0.9 N/tex, the strongest 4.1 N/tex, which while not typical, exceeds the best commercially available material and attests to the considerable promise of carbon nanotube fibres. Post spinning treatments such as annealing and twisting, have also been shown to produce some improvement in properties but not yet of comparable magnitude. The fibres wound-up in extreme conditions also show enhanced stiffness but reduced strain to fracture. Microscopy shows no difference to the bundles of collapsed nanotubes, although there is evidence that the bundles are better aligned. Work is in progress to quantify the changes in preferred orientation.
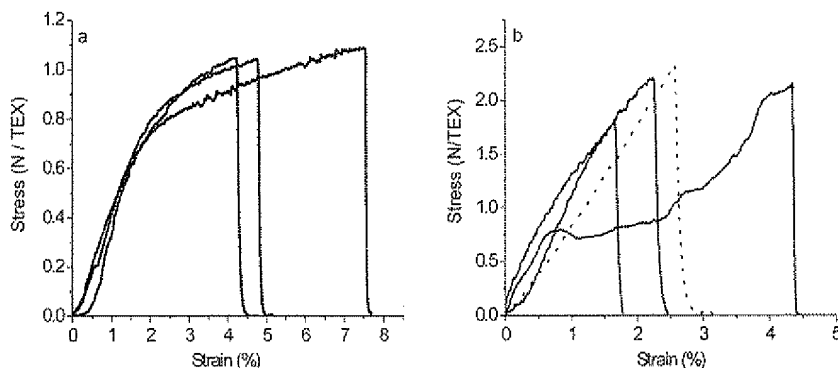

Figure 4. a) Stress strain curves of three fibres continuously wound from using the conditions listed in Table 3 (10 m min^{-1}). The curves are characterised by elastic and plastic regions. The data is 'raw' having been subject only to the calculation of specific stress by dividing the load by the tex of the fibre in the unstrained condition, and the calculation of strain from the extension using the original gauge length. The average values for the mechanical parameters from these three curves are: Strength: 1.1 N/tex. Stiffness: 60 N/tex and energy absorbed prior to fracture: 42 J g^{-1}. b) Stress strain curves of fibres wound from the furnace at maximum possible speed to extend the sock in situ (solid curves). The fibres were made under exactly the same furnace conditions as the standard material (4a). The average values for the mechanical parameters from these three curves are: Strength: 2.1 N/tex. Stiffness: 142 N/tex and energy absorbed prior to fracture: 32 J g^{-1}. The dotted curve on the plot corresponds to a carbon fibre (Grafil® 34-700) tested under identical conditions.

Also plotted on Figure 4b is the stress strain curve of a commercial carbon fibre (Grafil® 34-700) tested under identical conditions.

Table 2 compares the data seen on the direct spun CNT fibres with properties of commercially available high performance fibres. There are two types of direct spun fibres, the standard tougher fibres and the stronger fibres spun under extreme wind-up conditions.

Table 2. Summary of mechanical properties and comparisons with commercially available high performance fibres.

	Strength [N/tex]	Stiffness [N/tex]	Energy to fracture [J/g]
Direct spun CNT fibre. Standard conditions (best values)	1.2	65	60
Direct spun CNT fibres. Extreme wind-up (best values)	2.2	160	46
Carbon fibre [a] (Grafil® 34-700.)	2.4	90	30
Aramid fibre (Kevlar® 49)	2.5	90	35
PBO fibre (Zylon® HM)	3.7	172	45
Gel spun PE (Dyneema® SK 75)	3.5	113	63

[a] Measured in our laboratory.

treme wind-up conditions. The fibre spun in extreme wind-up conditions, has properties which would commend it as a high performance fibre with exceptional strength, stiffness and energy absorbing capacity. It is a 'yarn-like' carbon fibre, with excellent toughness. It should be emphasised that in many instances better properties than those quoted for the commercial products have been obtained under laboratory conditions.

The process of direct spinning of carbon nanotube fibres from the CVD reaction zone is yielding fibres with distinctly encouraging mechanical properties. While the best strength (2.2 N/tex) and stiffness (160 N/tex) promise competition for established carbon fibres, the maximum energy absorbed at fracture (46 J g^{-1}) is somewhat higher. The strong and stiff fibres consist mainly of very long double wall nanotubes of surprisingly large diameter in the 5–10 nm range. In line with theoretical predictions, these tubes have collapsed to give a dog-bone cross section. The flattened graphene layers form stacks, which account for the strong 002 reflection seen in the diffraction patterns.

While it might be expected that the individual nanotubes are strong and stiff in their axial direction, the translation of these properties to a fibre depends on stress transfer in shear between neighbouring tubes. To achieve good properties the tubes need not only to be long, but they need to share maximum contact area. In this respect stacks of flattened nanotubes are advantageous compared with touching round tubes, which may be somewhat polygonised. The synthesis of long, double wall nanotubes of unusually large diameter is therefore seen as one key to achieving good fibre properties. The aspects of the process, which produce such tubes, would appear to be the combination of comparatively large iron catalyst particles and the addition of sulphur, which seems to en-

sure assembly of the thin walled nanotubes on the catalyst particle surface, rather than multiwall nanotubes, requiring bulk rather than surface diffusion of the carbon. Work is underway to elucidate the exact mechanism further.

The cross-sectional structure of the bundles shown in Figure 2 is at first sight reminiscent of the meanderings of the folded graphene sheets in a carbon fibre structure. It is thus pertinent to ask what advantages direct-spun CNT fibres might have. The first is their highly encouraging energy to fracture coupled with stress-strain curves showing a yield stress followed by plastic deformation, rather reminiscent of a ductile metal, but at higher stress levels. The origin of the toughness is likely to be the sliding of nanotube bundles past each other over large distances in comparison with their diameters. The second is the fact that the structure is engineered 'bottom up' rather than 'top down' as is the case of PAN-based carbon fibres. This provides greater opportunity for control of the nanotube structure, their assembly into bundles and then into fibres. The third significant factor is that the direct spinning process is intrinsically simple: liquid hydrocarbon feedstock containing catalyst is converted into the wound up product continuously in a single reactor. This simplicity means that production costs can be lower than for existing high-performance fibres.

Experimental

The process for the direct spinning of carbon nanotube fibres from the CVD reaction zone has already been described [5]. It is diagrammatically depicted in Figure S1. Table 3 in the Supporting Information summarises the experimental conditions for the production of the fibres described in this paper.

The direct spinning process makes carbon nanotubes from hydrocarbon feedstock and then spins them into high performance fibres in a single operation. A liquid hydrocarbon feedstock (ethanol or hexane) containing thiophene and ferrocene (Fe/S = 3.5) is carried by hydrogen into a furnace held, typically, at 1300 °C. In the furnace, the ferrocene decomposes into a 'mist' of nano-sized iron catalyst particles over which the feedstock cracks to grow the nanotubes. A wide range of feedstocks work, including ethanol, which is derivable from renewable resources. Processing conditions can be varied to favour the formation of either single- or multiwall carbon nanotubes. A key to the process is that the nanotubes are very long and are made extraordinarily fast so that they entangle in the hot zone to form an aerogel. The fibres (Fig. S2a) are then spun from the aerogel and wound continuously onto a spool placed outside of the furnace (Fig. S2b).

It is important to appreciate that successful spinning can occur over a range of conditions, and a variety of feedstocks. The conditions described in Table 3 are defined as "standard" because they are in the centre of the spinnability range. For instance, the process can be operated successfully with temperatures ranging from 1100 to 1500 °C, feedstock injection rates from 2 to 12 ml h^{-1} and carrier gas flow rates from 400 to 4000 ml min^{-1}.

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